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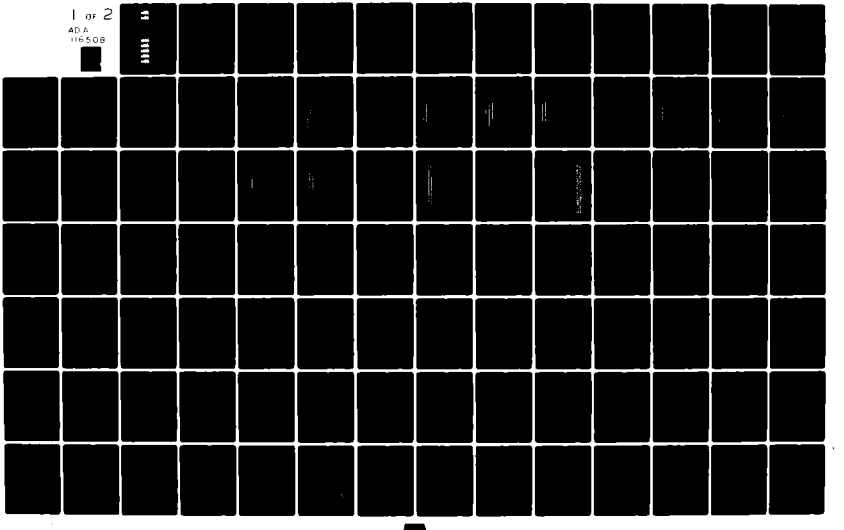

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Mechanical Technology Incorporated

Research and Development Division

(12)

FINAL REPORT

ANALYTICAL FERROGRAPHY STANDARDIZATION

Prepared For:

OFFICE OF NAVAL RESEARCH
ARLINGTON, VIRGINIA 22217

CONTRACT N00014-81-C-0012

JANUARY 1982

Final Report

MTI Technical Report No. 82TR56

ANALYTICAL FERROGRAPHY STANDARDIZATION

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A. S. Maciejewski

Applications Engineering
Mechanical Technology Incorporated
Annapolis, Maryland

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Wear particle technology is a recent development in the equipment wear field. This technology is based on the analysis of wear debris as a nondestructive reflection of the surface wear condition of the respective monitored wear process. Such a monitoring approach can be applied to everything from simple wear testing to sophisticated multicomponent wear systems. Wear particle analysis technology is rapidly establishing itself as a valuable tool in both the wear prevention and wear control arenas.

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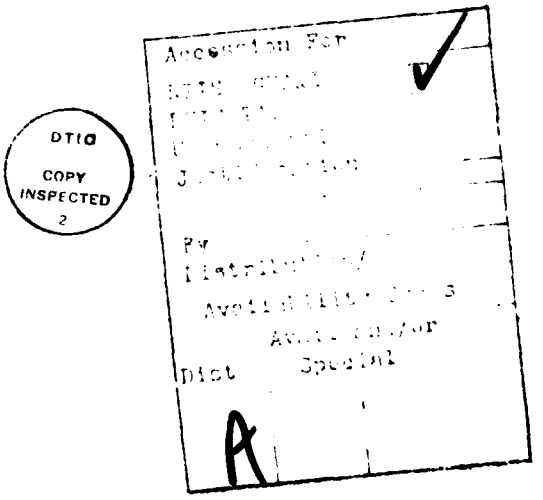

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FORWARD

This report presents the results of a study conducted by Mechanical Technology Incorporated (MTI) for the Office of Naval Research (ONR) under Contract N00014-81-C-0012. This study was based on the standardization of Analytical Ferrography.

The work was performed under the direction of Lt. Cdmr. H. Martin and Mr. M. K. Ellingsworth, Navy Program Managers. Mr. Peter Senholzi was the Program Manager for the contract at MTI, with Mr. Alan Maciejewski serving as Project Engineer.

The authors wish to acknowledge the program assistance provided by Mr. H. Martin, Mr. R. Miller, and Mr. M. K. Ellingsworth of the Navy. Appreciation is also extended to personnel from Oklahoma State University, Foxboro Analytical, Michigan Technological University, National Bureau of Standards, Joint Oil Analysis Program Technical Support Center, and the Naval Air Engineering Center for their program participation and extensive contributions.

ABSTRACT

Wear particle technology is a recent development in the equipment wear field. This technology is based on the analysis of wear debris as a nondestructive reflection of the surface wear condition of the respective monitored wear process. Such a monitoring approach can be applied to everything from simple wear testing to sophisticated multicomponent wear systems. Wear particle analysis technology is rapidly establishing itself as a valuable tool in both the wear prevention and wear control arenas.

Analytical Ferrographic analysis is a relatively new approach to the analysis of wear debris. Until recently, this technique has been utilized as a research tool in a limited number of laboratory facilities. However, as a result of initial successful utilization, Ferrographic technology is receiving ever increasing interest. This increasing interest level has raised serious questions with respect to standardization and repeatability.

This report describes an effort to quantify and apportion Analytical Ferrography repeatability/nonrepeatability. Under a program sponsored by the Office of Naval Research, six leading laboratories contributed controlled Analytical Ferrographic analysis data. This data has been analyzed and the resulting repeatability/nonrepeatability assessed with respect to analysis variables.

1.0 INTRODUCTION

Mechanical system availability, efficiency, and life are functions of both structural integrity and wear integrity. Emphasis to date has been placed on structural integrity, with a "throw away" philosophy accommodating the consequences of low wear integrity. Recent resource limitations, however, have prompted substantial interest into the area of wear integrity optimization. This optimization process is approached through the interdisciplinary technology of tribology. Tribological technology involves both the wear aspects of prevention and control. Wear prevention occurs primarily in the equipment design process, while wear control is primarily instituted in the operational arena.

Wear particle technology is a relatively recent development in the equipment wear field. This technology is based on the analysis of wear debris as a nondestructive reflection of the surface wear condition of the respective monitored wear process. Such a monitoring approach can be applied to everything from simple wear testing to sophisticated multicomponent wear systems. Wear particle analysis technology is rapidly establishing itself as a valuable tool in both the wear prevention and wear control arenas. In order to fully realize the potential of wear particle analysis technology, additional research efforts must be implemented in order to enhance and expand the technology.

Analytical Ferrographic analysis is a relatively new approach to the analysis of wear debris. Until recently, this technique has been utilized as a research tool in a limited number of laboratory facilities. However, as a result of initial successful utilization, Ferrographic technology is receiving increasing interest. This increasing interest level has raised serious questions with respect to standardization and repeatability.

This program deals with the standardization of Analytical Ferrography. Primary emphasis is directed at the quantification of Ferrographic repeatability, variation apportionment between respective significant contributing factors, and the development of a standardized procedure.

2.0 BACKGROUND

Analytical Ferrography is based on the magnetic precipitation and subsequent analysis of wear debris from a lubricant sample. The approach utilized involves passing a volume of lubricant over a glass substrate which is supported over a magnetic field. Permanent magnets are arranged in such a way as to create a varying field strength over the length of the substrate. This varying strength results in the precipitation of wear debris (magnetic and ferromagnetic) in a distribution with respect to size/mass over the substrate length (approximately 55 mm). Once rinsed and fixed to the substrate, this deposit serves as an excellent media for optical analysis of the composite wear particulates.

Ferrographic substrate deposit analysis involves the characterization of debris quantity, distribution, elemental composition, and morphology. This total analysis effort involves both quantitative and qualitative assessments.

Quantitative assessments are derived for quantity and size distribution characterization utilizing a light reflected/light transmitted type densitometer. These assessments are registered by indicating the percentage of blocked area in a particular microscopic field of view. Readings are taken over the length of the substrate deposit in order to characterize debris size distribution.

Elemental composition and morphological debris assessments are very qualitative in nature. They involve the manual characterization of debris deposits relying on observations conducted through an optical microscope.

This deposition and assessment process involves a multitude of variables. Such aspects as sample preparation, sample dilution, sample volume, sample viscosity, debris concentration, densitometer type, densitometer calibration, measurement approach, measurement indexing, and debris distribution, all affect Ferrographic assessment results. These aspects can be generally categorized in three groups:

- technique/procedure,
- equipment, and
- operator.

In the initial stages of Ferrographic analysis, it was left up to each individual laboratory to address the numerous analysis variables.

2.1 The Technical Cooperative Program

During the mid 1970's, an international committee was established under The Technical Cooperative Program (TTCP) with the objective of fostering equipment health monitoring. A prime emphasis area of this committee was wear particle analysis and specifically Ferrographic Analysis. As part of this committee's efforts, lubricant samples were distributed among participating laboratories, for wear debris analysis. Upon comparing sample analyses, it became apparent that slide variations existed between laboratory results. Further investigations revealed that at least a portion of this variation was due to individually developed/tailored Ferrographic procedures. No variation apportionment could be made, however, between procedure, operator, and equipment. Thus, a significant standardization and repeatability problem was identified. This problem was intensified by the fact that Ferrography was being applied by an ever increasing number of organizations in a variety of applications.

2.2 Navy Standardized Procedures

As a first step in attacking the problems of Ferrographic standardization, the Navy proceeded to generate a preliminary detailed Ferrographic Procedure. Due to procedural controversies, dual procedural approaches were included for density lighting technique and density reading indexing approaches.

In order to verify this procedure, clarify controversial dual approaches, quantify repeatability, and identify respective significant contributing factors, a "round robin" sample analysis exercise was established between six major wear particle analysis facilities.

2.2.1 Verification Program

In order to verify the Preliminary Navy Analytical Ferrography Standardization Procedure, a joint program was developed and supported by six laboratories;

Michigan Technological University, Oklahoma State University, Foxboro Analytical, the Naval Air Engineering Center, National Bureau of Standards, and the Joint Oil Analysis Program Technical Support Center. Sets of identical lubricant samples were distributed to each laboratory for analysis. Sample variables consisted of lubricant type and debris concentration level. Laboratories were directed to generate Ferrogram slides from each samples as per the preliminary standardized procedure. Each slide was to be analyzed using all combinations of suggested indexing and lighting techniques.

In conjunction with this effort, sample preparation variables were to be eliminated by the circulation of sets of pre-made slides between each of the laboratories. Each laboratory was to analyze the pre-made sets in the same manner as outlined above.

Respective data has been generated by these six organizations and has been subjected to an in-depth statistical treatment to be summarized in the following discussions. This analysis was limited to Analytical Ferrography and did not include either Direct Reading or In-Line Ferrographic Techniques.

3.0 TECHNICAL APPROACH

In order to adequately understand the statistical analysis of the verification program, discussions of the pertinent variables, program organization, and statistical tools will be presented.

3.1 Analysis Variables

As mentioned above, in order to verify the Preliminary Navy Analytical Ferrography Standardized Procedure, a joint program was developed and supported by several leading wear debris laboratories. A two phase "round robin" approach was instituted. Each phase consisted of the analysis of fluid samples as well as the analysis of pre-made Ferrograph slides.

Primary analysis variables addressed under this "round robin" exercise were procedure, equipment, and operator/location. Secondary addressed variables included; fluid type, debris concentration, debris size distribution, Ferroscopic type, Ferrogram slide location, microscope lighting approach, and slide indexing approach. Round robin test design incorporated both primary and secondary variable treatment.

3.2 Program Organization

The Naval Ferrography Verification Program was initiated in 1979. Under the direction of the Office of Naval Research (ONR) Scientific Officer Lt. Cdmr. H. Martin, the Naval Air Engineering Center (NAEC) was tasked to plan, organize, and control a coordinated laboratory sample analysis effort, evaluating the precision and repeatability of the Analytical Ferrographic equipment and respective procedure. Four facilities were initially selected to participate in this effort. All were experienced in the Ferrography methodology and possessed the desired technical expertise and professionalism to adequately perform in the exercise. The four initial facilities and their representatives include:

1. Foxboro Analytical (FOX) - D. Anderson
2. Michigan Technological University (MTU) - Dr. J. Johnson
3. Naval Air Engineering Center (NAEC) - P. Senholzi
4. Oklahoma State University (OSU) - Dr. E. Fitch

After completion of Phase I of this program, a statistical analysis was performed on the data generated. From this analysis certain preliminary conclusions were made. With the aid of these preliminary findings, ONR authorized Mechanical Technology Incorporated (MTI) to plan, organize, and direct Phase II of the verification program.

In addition to the four initial participants, two other organizations were included in Phase II of the program. These organizations were:

1. Joint Oil Analysis Program Technical Support Center (JOAP-TSC) - R. Lee
2. DOC National Bureau of Standards (NBS) - Dr. W. Ruff

At the conclusion of Phase II all data (including Phase I) were thoroughly analyzed. In addition, a draft standardized procedure for Analytical Ferrography was developed from this exercise in order to minimize error in technique applications. As stated previously, the Naval Ferrography Verification Program was performed in two separate phases. The following is a description of the organization of each phase.

3.2.1 Phase I Organization

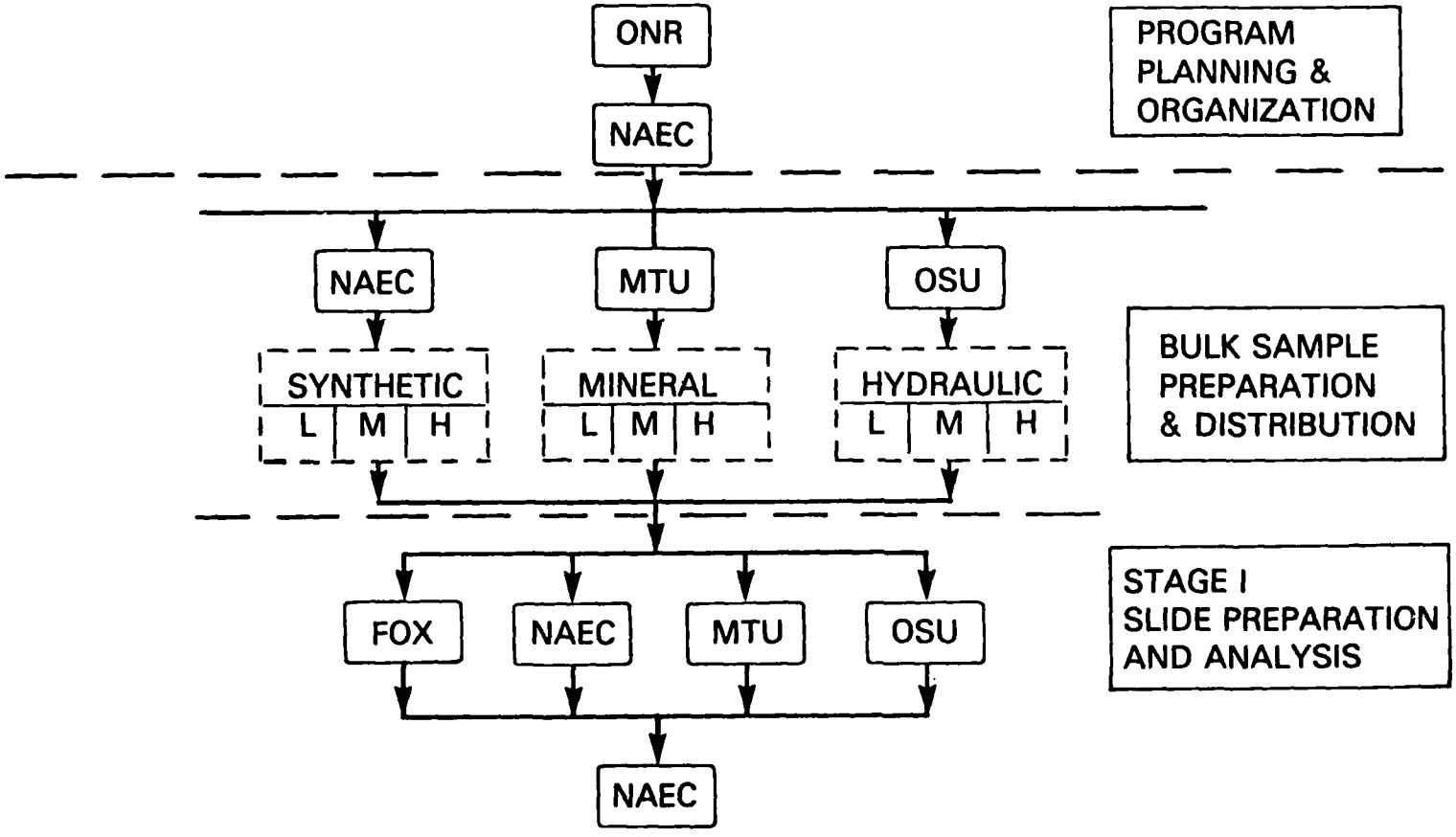
The initial phase of the program was directed and controlled by NAEC. This phase was divided into a sample analysis stage and a slide analysis stage. Figure 1 illustrates the program organization during Stage I of this phase. The three facilities responsible for providing fluid samples were selected on the basis of their expertise. The three facilities, NAEC, MTU, and OSU, provided three different types of fluids; synthetic lubricant, mineral lubricant, and mineral-based hydraulic fluid, respectively. For each fluid type, the facility provided sets of three samples containing wear debris of low, medium, and heavy concentrations.

Each sample generating facility then distributed a complete set of their respective fluid type samples to each of the participating analysis facilities, while retaining a set for their analysis.

FIGURE 1

PHASE I STAGE I

FERROGRAPHY VERIFICATION FLOW CHART



After receiving the three sets of fluid samples, each facility was to prepare ferrograms of each sample type/concentration, according to a set of general guidelines. Figure 2 illustrates the sample preparation and analysis activity during Stage I. For each laboratory, three Ferrogram slides were made from each sample bottle (nine total sample bottles). For each Ferrogram slide made, density reading sets were obtained four times. As a result, each laboratory produced 27 Ferrograms and 108 reading sets. When including all facilities, this amounted to a total of 108 Ferrograms and 432 sets of readings.

After Stage I was completed, each facility was instructed to assemble a set of four pre-made slides. The selected slide sets were first read by the generating laboratory and then packaged and distributed for analysis as indicated in Figure 3. Each facility read each slide four times, as in Stage I, and forwarded the set to the next facility. The slide sets were, at the conclusion of the analysis sequence, returned to the respective generating facility in order to verify that the slides did not degrade during transit. At the end of this stage, a total of 320 reading sets were accomplished. The final breakdown by fluid types for Stage II is summarized in Figure 4.

3.2.2 Phase II Organization

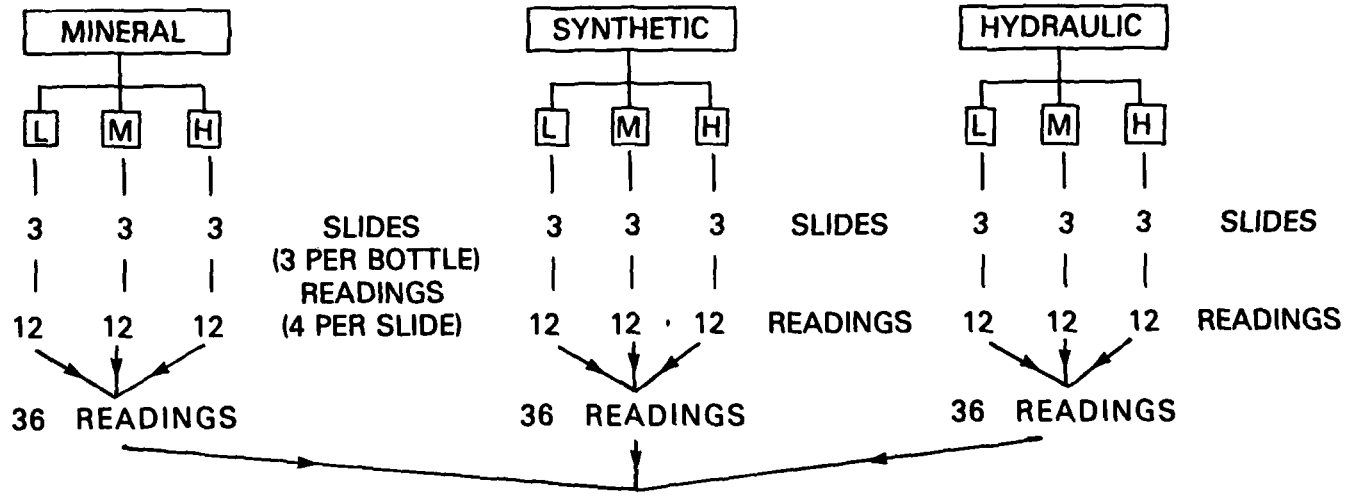
The second phase of the program was organized, directed, and controlled by MTI. Based upon a detailed review of the data generated in the first phase, certain portions of the initial program phase were revised or eliminated. With the introduction of Phase II, a Ferrography procedure was provided for utilization in the performance of the sample analysis as well as for solicitation of critical comment.

As noted previously, two additional facilities were added to the study during this second phase. As opposed to the first phase, sample distribution was centrally coordinated by MTI. Three labs were solicited for bulk fluid samples, as under Phase I, based on their experience with a particular fluid. From MTU, NBS, and OSU, MTI received mineral lubricant, synthetic lubricant, and mineral-based hydraulic fluid, respectively. Bulk fluid types were divided into six sets of three bottles each, and subsequently distributed to the participating

FIGURE 2

PHASE I — STAGE I
ANALYSIS SUMMARY

- LABORATORY



- SAMPLE FLUID TYPE AND CONCENTRATION

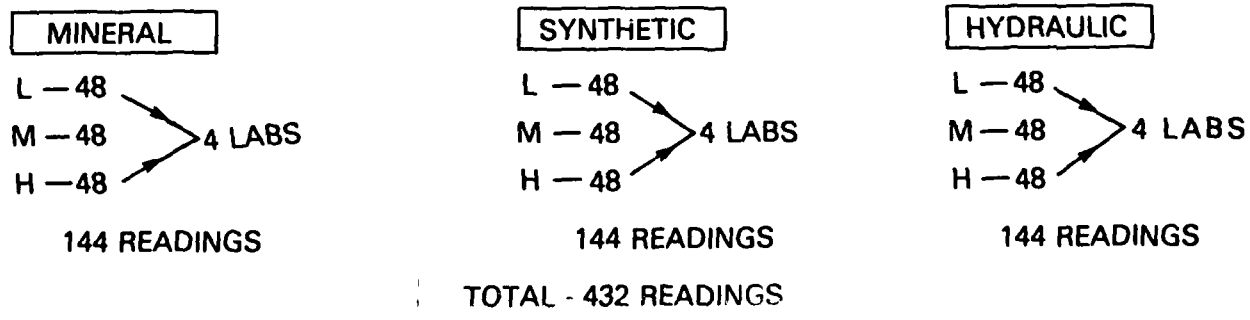


FIGURE 3

PHASE I STAGE II

FERROGRAPHY VERIFICATION FLOW CHART

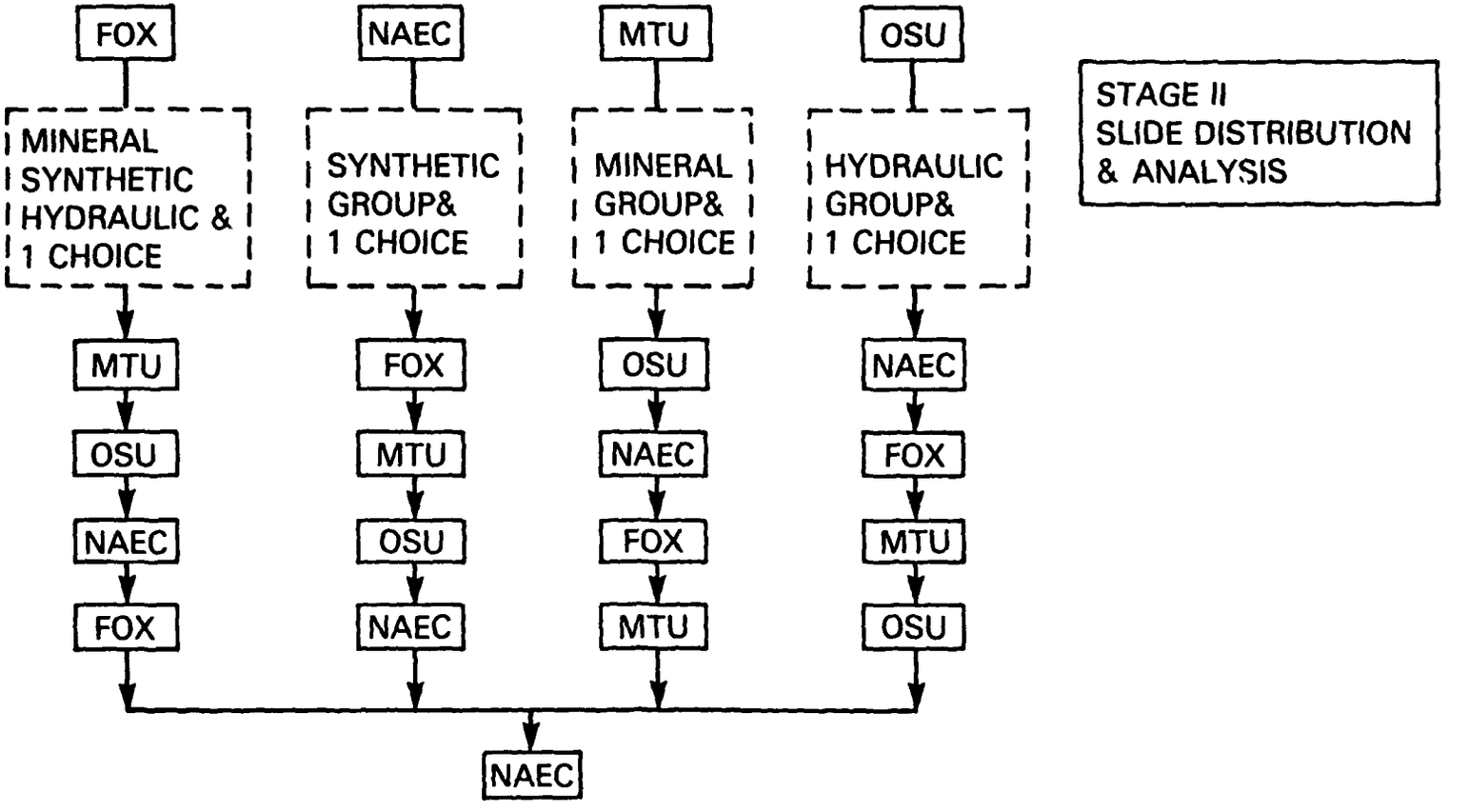


FIGURE 4

PHASE I — STAGE II
ANALYSES SUMMARY

- LABORATORY

FOX	NAEC	MTU	OSU
MIN-LT — 4	SYN-LT — 4	MIN-LT — 4	HYD-LT — 4
SYN-MED — 4	SYN-MED — 4	MIN-MED — 4	HYD-MED — 4
SYN-HVY — 4	SYN-HVY — 4	MIN-HVY — 4	HYD-HVY — 4
HYD-HVY — 4	HYD-HVY — 4	SYN-HVY — 4	SYN-HVY — 4
16 READINGS	16 READINGS	16 READINGS	16 READINGS

MTU 16	FOX 16	OSU 16	NAEC 16
OSU 16	MTU 16	NAEC 16	FOX 16
NAEC 16	OSU 16	FOX 16	MTU 16
FOX 16	NAEC 16	MTU 16	OSU 16
80 READINGS	80 READINGS	80 READINGS	80 READINGS

TOTAL - 320 READINGS

- SAMPLE FLUID TYPE AND CONCENTRATION

MINERAL	SYNTHETIC	HYDRAULIC
LT— 40	LT— 20	LT— 20
MED— 20	MED— 40	MED— 20
HVY— 20 → 4 LABS	HVY— 80 → 4 LABS	HVY— 60 → 4 LABS
80 READINGS	140 READINGS	100 READINGS

TOTAL - 320 READINGS

facilities, as illustrated in Figure 5. Upon receipt of the sample set, each facility was instructed to prepare five Ferrogram slides from each sample bottle for a total of fifteen Ferrograms per facility. For each of the fifteen Ferrograms, five sequential reading sets were obtained, for a total of 75 reading sets per facility. Figure 6 illustrates the output per facility as well as the final tabulation by sample fluid type.

The data from Stage I of the second phase was then forwarded to MTI. This data was statistically analyzed and presented at an interim review meeting of the participants. At this meeting the participants also submitted their respective Phase II, Stage I slides to MTI for distribution in Stage II of the program. Participant comments and recommendations concerning the preliminary standardized procedure were presented during this meeting.

Stage II of the second phase involved the distribution of a slide set, assembled by MTI. This set consisted of two Ferrogram slides of each fluid type for a total of six slides. The slides were marked with a code number so as to avoid biasing the results of the participating laboratory. This slide set was sent out by MTI to the first facility for analysis. Figure 7 indicates the routing procedure for the slide set and corresponding results. For each slide, five sequential reading sets were performed by each laboratory. This resulted in 180 total reading sets or 60 reading sets per fluid type as summarized in Figure 8.

Upon receipt of all data, MTI performed a statistical analysis and compared the results to the previous program stages. In addition to the statistical analysis, an interim draft of the Navy Analytical Ferrography Standardized Procedure was developed.

3.3 Statistical Approach

Respective Ferrographic analysis data were submitted to a central location from each participating laboratory for each round robin phase. This data consisted of sets of density readings for each sample/slide, indexed with respect to Ferrogram slide location. Supporting information for each set was

FIGURE 5

PHASE II STAGE I

FERROGRAPHY VERIFICATION FLOW CHART

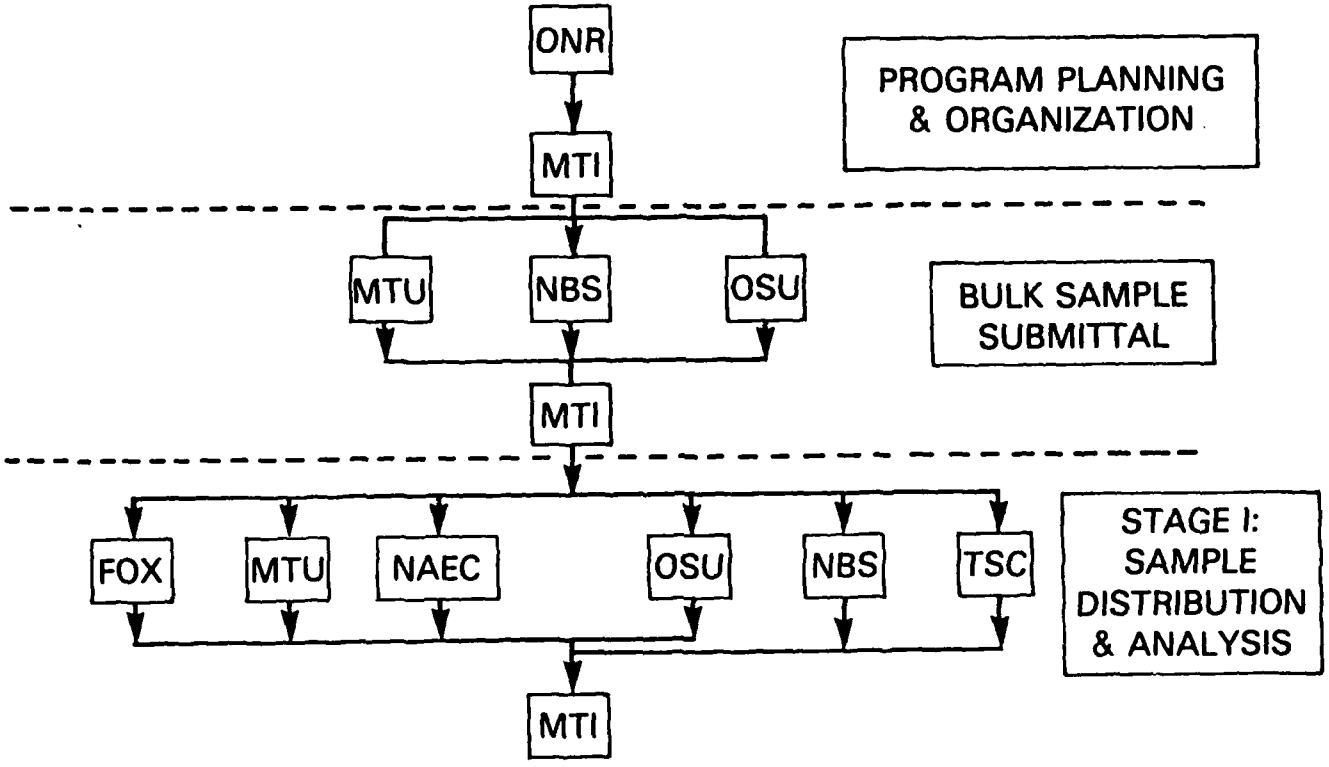
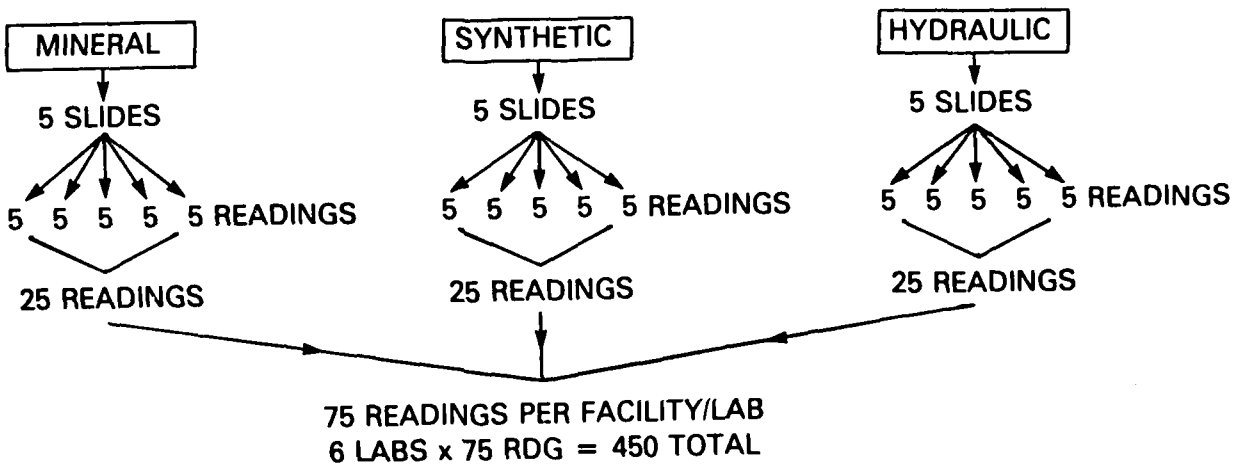


FIGURE 6

PHASE II — STAGE 1 SUMMARY

ANALYSES SUMMARY

- LABORATORY:



- SAMPLE FLUID TYPE

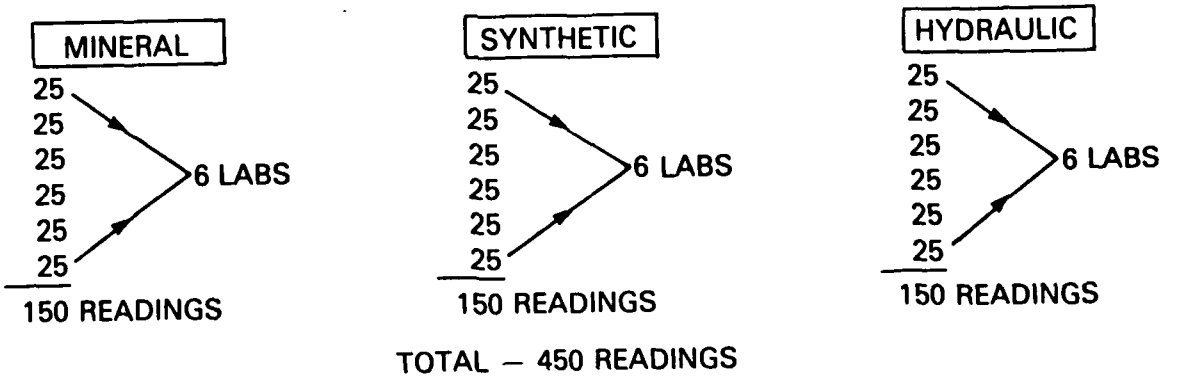


FIGURE 7

PHASE II — STAGE II SLIDE DISTRIBUTION AND ANALYSIS

SLIDE SET	# OF READINGS
2 MINERAL	10 (5 Each)
2 SYNTHETIC	10 (5 Each)
2 HYDRAULIC	10 (5 Each)

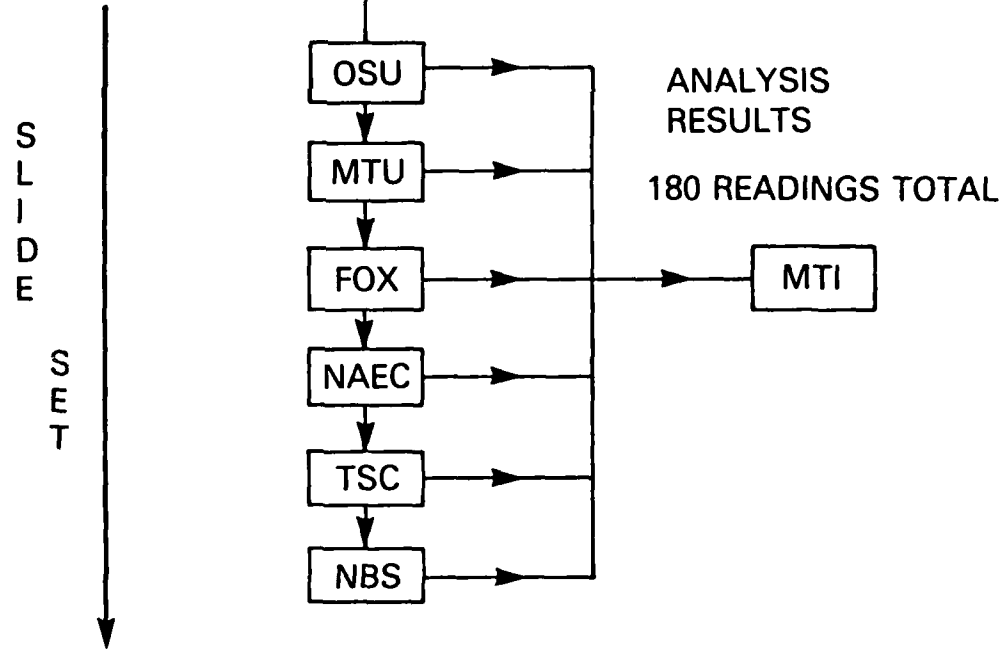
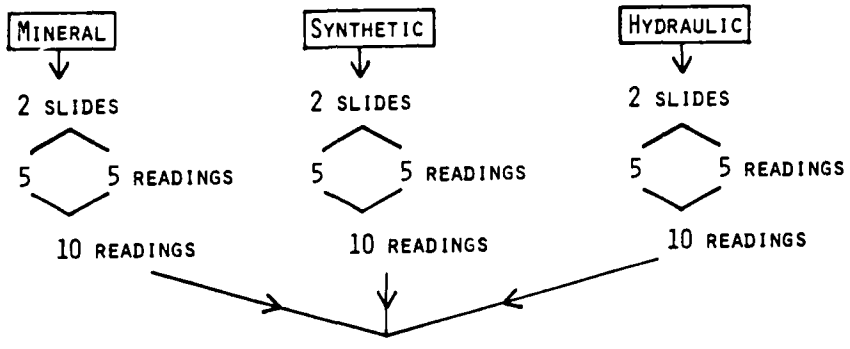


FIGURE 8

PHASE II - STAGE II SUMMARY

ANALYSES SUMMARY

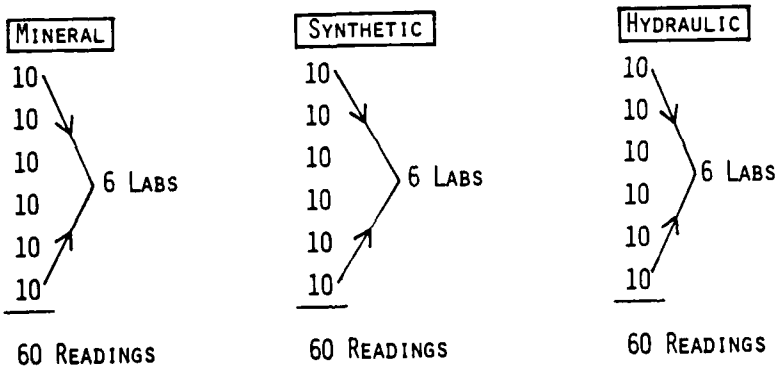
- LABORATORY



30 READINGS PER FACILITY/LAB

6 LABS X 30 READINGS = 180 TOTAL

- SAMPLE FLUID TYPE



TOTAL - 180 READINGS

also submitted which included such items as volume of fluid analyzed, dilution ratio, lighting approach, indexing approach, operator, and sample number. Statistical analyses were performed on these data sets in order to determine repeatability as well as apportion nonrepeatability among the competing significant variables.

A four facet statistical format was applied to this data base. These four facets included data plots (slide position versus normalized density), analysis of variance, coefficient of variation analysis, and a graphical regression analysis.

3.3.1 Data Plots

Data plots and supporting analysis included the following elements:

- Plots: normalized density readings versus slide position
- Lease Squares Line: $Y = ax + b$
- *Mean Value of Density:* YBAR
- Standard Deviation of Density Distribution: SY
- Standard Error of the Estimate: SYX

  This quantity is a measure of the scatter of points around the least squares line. It is also an estimate of the standard deviation of the y population for a given x. The definition is:

$$S_{yx} = \sqrt{\frac{\sum_{i=1}^{n} (y_i - y_{ic})^2}{n-2}}$$

  where y_i = actual values of y

  y_{ic} = values of y computed from the equation of the line

  n = number of points.

  So, the more scatter the larger S_{yx} is going to be.

- Correlation Coefficient: r

  This is a measure of the quantitative association between variables.

- Normal distribution graphs:

  All the readings from each category (e.g. each lab) were treated, and the mean and standard deviation were calculated. These numbers were then substituted into the equation of a normal distribution:

$$f(x) = \frac{1}{S_x \sqrt{2\pi}} e^{\frac{-(x-\bar{x})^2}{2S_x^2}}$$

  where x is the mean and S_x is the standard deviation.

  Then the curves for all members of one category (e.g. all labs) were superimposed. The axis of symmetry of each curve is at the mean value, while the width of each curve is a function of the standard deviation. The wider the curve, the more scatter there was in the data.

  A normal curve can also be interpreted like a histogram, e.g. frequency as a funtion of value. Hence a short squat curve would result from a wide range of values occurring at about the same frequency.

- Comparison Output:

  The various factors in each category were compared as follows: the mean and standard deviation for all readings from factor 1 (e.g. Lab 1) were calculated, as were the same quantities from factor 2 (e.g. Lab 2).

  There are standard statistical tests to determine, with a certain degree of confidence, whether one can claim that the mean from factor 1 readings is significantly different from the mean from the factor 2 readings, and likewise for standard deviation.

From these tests it was possible to claim with 95% confidence used for example, that the mean of readings from Lab 1 is significantly different from the mean of readings from Lab 2, and further, a range of that diference can be calculated. We achieve an expression of the form:

$(\overline{X}_1, \overline{X}_2$ are respective means; A, B are calculated.)

and this is what appears in the output.

For standard deviation, the expression involves a quotient:

$$A \leq \frac{S_1}{S_2} \leq B$$

(S_1, S_2 are respective s.d.; A, B are calculated), so the output indicates that one standard deviation differs by a certain factor from the other.

An example of the data plot is presented in Figure 9.

3.3.2 Analysis of Variance

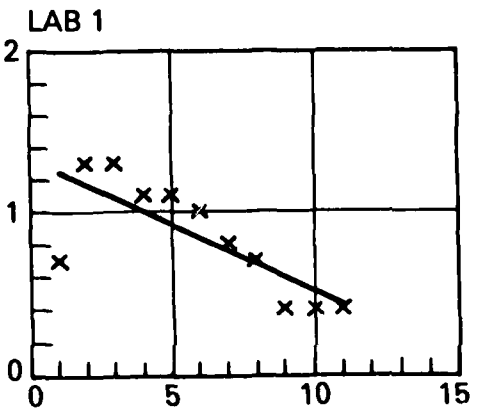
When several sources of variation are acting simultaneously on a set of variations, the variance of the observations is the sum of the variance of the independent sources. So, the total variance would be the sum of the variance due to each independent factor, plus the remaining variance (called the residual) due to randomness. The variance of each factor, or group of factors, is compared to the residual variance, and this quotient is compared with a table value (F test) to tell whether a significant difference exists between factors. Figure 10 represents an example of analysis of variance.

3.3.3 Coefficient of Variation Analysis

The coefficient of variation serves to reflect variability of a population. This statistical treatment included the following elements:

FIGURE 9

DATA PLOTS



MTI
PH887

FERROGRAPHY DATA

DENSITY VS SLIDE POSITION
LAB 1 SYNTHETIC
LIGHT
TRANSMITTED
NEW METHOD

$Y = -0.081\ X + 1.322$

YBAR = 0.836 SY = 0.347

SYX = 0.232 R = 0.743

Y = LEAST SQUARED EQUATION
YBAR = AVERAGE DENSITY READING
SY = STANDARD DEVIATION
SYX = STANDARD ERROR OF ESTIMATE
R = CORRELATION COEFFICIENT

FIGURE 10

ANALYSIS OF VARIANCE
FERROGRAPHY DATA

SOURCE OF VARIATION	SUM OF SQUARES SS	DEGREES OF FREEDOM DF	MEAN SQUARE MS/DF	MEAN-SQUARE RATIO MSR MS/MSRESID	F VALUE 90 PERCENT CONFIDENCE	CONCLUSION
TYPE OF OIL	2.	0.7970E+05	0.3985E+05	0.1093E+05	2.30	SIGNIFICANTLY EFFECTS DENSITY
CONCENTRATION	2.	0.2215E+05	0.1107E+05	0.3038E+04	2.30	SIGNIFICANTLY EFFECTS DENSITY
LABORATORY	4.	0.1592E+05	0.3980E+04	0.1092E+04	1.94	SIGNIFICANTLY EFFECTS DENSITY
LIGHTING	1.	0.5269E+03	0.5269E+03	0.1445E+03	2.71	SIGNIFICANTLY EFFECTS DENSITY
METHOD	1.	0.1838E+03	0.1838E+03	0.5041E+02	2.71	SIGNIFICANTLY EFFECTS DENSITY
SLIDE POSITION	10.	0.7250E+02	0.7250E+01	0.1989E+01	1.60	SIGNIFICANTLY EFFECTS DENSITY

- Mean Value: YBAR
- Standard Deviation: SY

$$SY = \sqrt{\Sigma \frac{(Y-\bar{Y})^2}{n-1}}$$

- Coefficient of Variation: COV

  (Percent standard deviation)

$$COV = \frac{SY}{\bar{Y}} \times 100$$

An example of coefficient of variation is presented in Figure 11.

3.3.4 Graphical Regression Analysis

The graphical regression analysis technique involves a comparison evaluation utilizing an "x-y"/45° plot of all possible respective data combinations. For example, in the case of the Ferrographic procedure verification Phase I, the possible combinations would consist of five data sets (two for NAEC) which results in ten possible combinations.

$$\begin{bmatrix} 5 \\ 2 \end{bmatrix} = \frac{5!}{2!(5-2)!} = 10$$

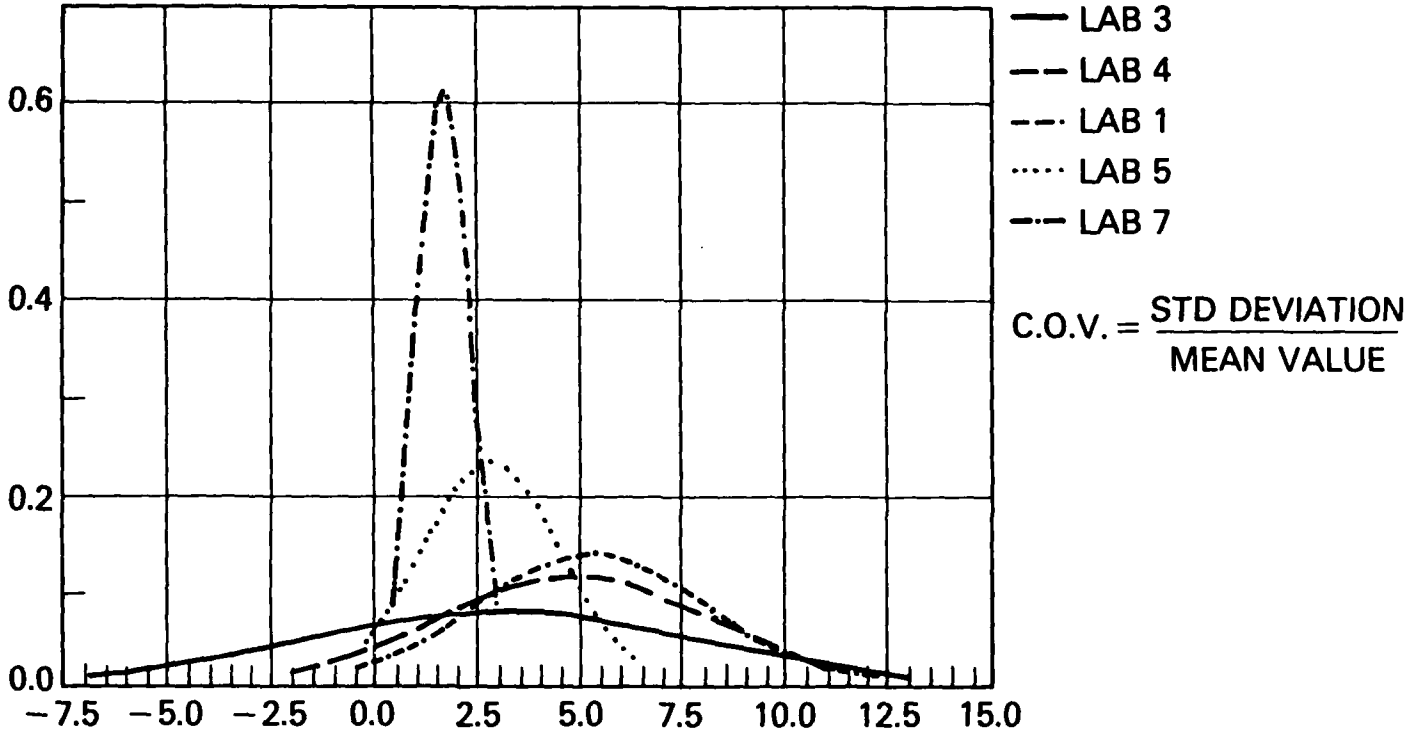
Three types of lubricants will result in (10 x 3) or 30 combinations. The three concentration levels of debris, two lighting techniques, and the two indexing approaches, will produce 360 possible combinations and thus 360 "x-y" comparison plots. Plots will also be made for circulated slide data.

Outputs from the resulting plots were a regression line fit, regression and correlation coefficients, and intercept. Evaluation of this data provides estimates of precision, accuracy, discrimination, and bias which are defined as follows:

- Precision is defined as the degree of repeatability of the measurements of the results taken at each measuring laboratory. It is affected by variables in instrumentation, personnel, handling, environments, etc. It can be designated as " ΔM": measurement error variation.

FIGURE 11

COEFFICIENTS OF VARIATION ANALYSIS
SAMPLE DISTRIBUTIONS & LABORATORIES



$$C.O.V. = \frac{\text{STD DEVIATION}}{\text{MEAN VALUE}}$$

- Accuracy is defined as a deviation, from the "true" value of a random reading due to biases and precision variation.

- Discrimination can be defined as the ratio of " ΔP" to " ΔM" where " ΔP" is sample-to-sample variation.

- Bias is defined as the average difference of measured values between laboratories.

These terms are graphically shown in Figure 12.

3.3.4.1 Example

The following example is provided in order to demonstrate the x-y technique.

Each of the pairs of standard alloys, 375 USR and 301 USR (U.S. Reduction), have known concentrations of each element, shown by the two arrows on the vertical axes on the following data plots.

Four to five samples were taken from the standard USR bar and analyzed by the client's metallurgical lab equipment. Those results, which evaluate how good or poor that equipment is, are shown on the horizontal axis labelled "Sample", Figure 13.

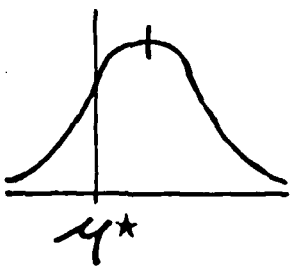
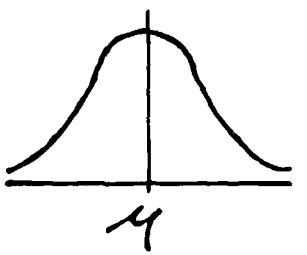
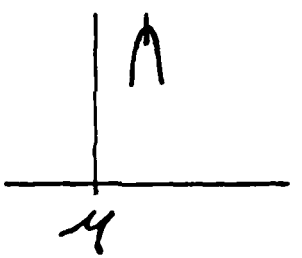
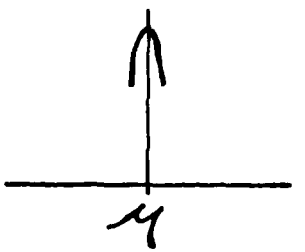
The Zn and Cr charts show no bias, and good discriminating ability. But Mn and Mg are biased; the lab equipment is reading too low. Fe and Si are biased the other way; the equipment is reading too high. The discrimination ratio for measuring Ti is inadequate, being close to a ratio of only 1/1.

3.3.4.2 Format

An example of the graphic evaluation format as applied to Ferrographic data is presented in Figure 14.

FIGURE 12

REPEATED READINGS

	BIASED	UNBIASED
IMPRECISE		
PRECISE		 Most accurate for single readings.

★ μ = TRUE VALUE

ACCURACY AS A FUNCTION OF REPEATABILITY TO A TRUE VALUE

FIGURE 13

USR STANDARD

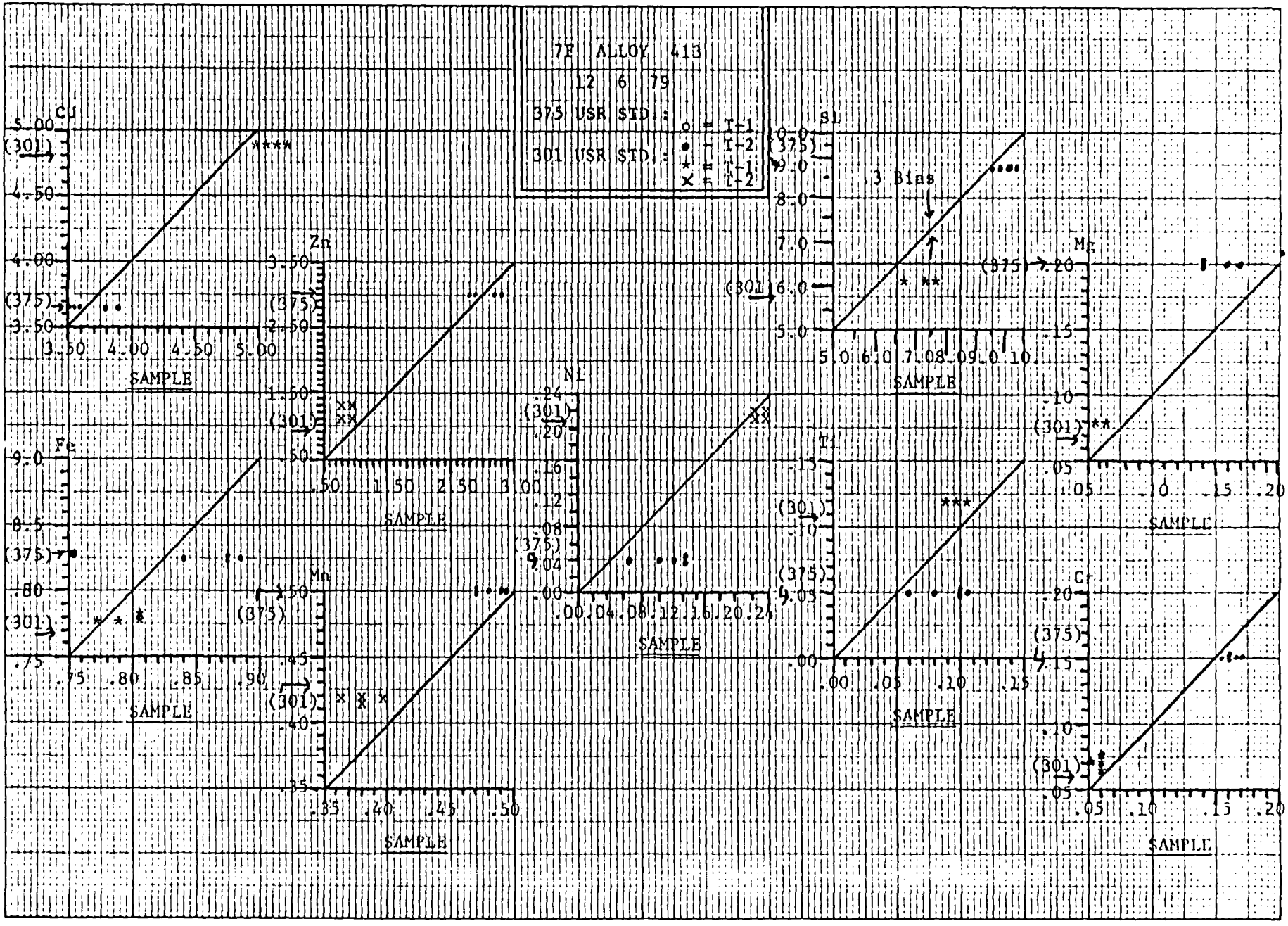
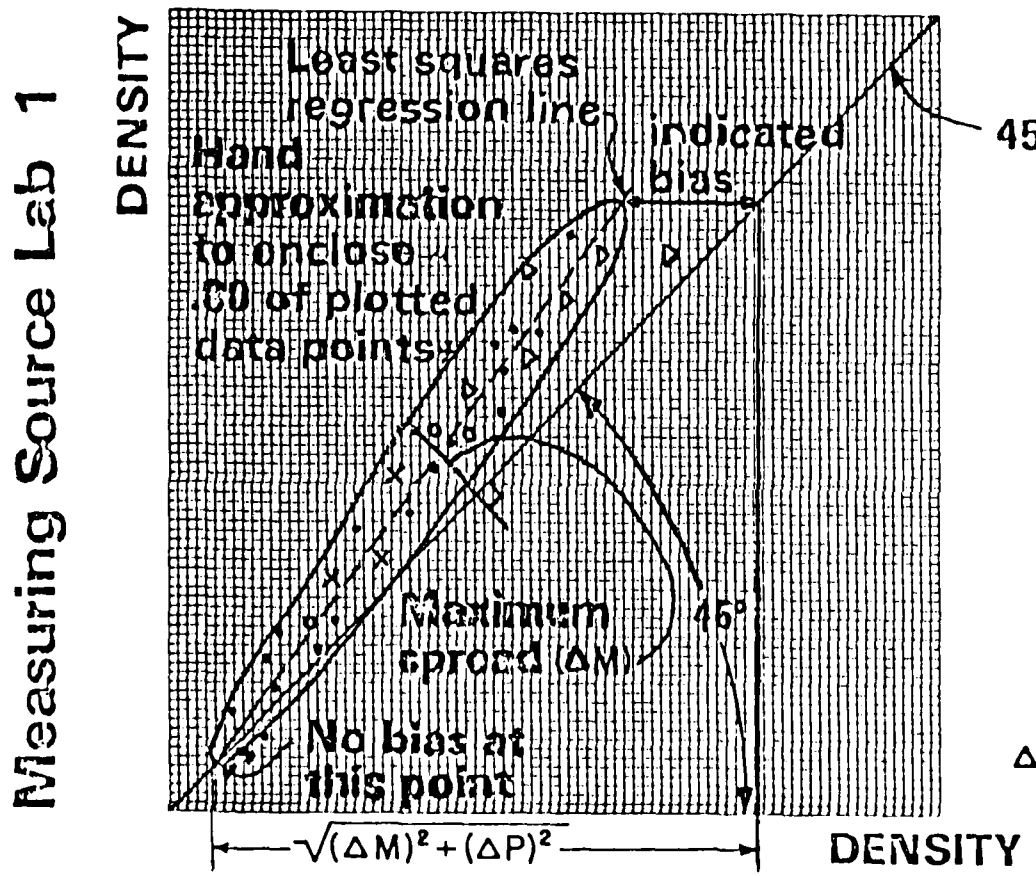


FIGURE 14

GRAPHICAL EVALUATION ANALYSIS (SAMPLE)



(For one oil type and density prepared at one source, and one measuring method)

Position Legend, (mm)

- • 0 mm
- × 20 mm
- ▷ 50 mm
- · Others

ΔP can be calculated

3.3.4.3 Illustration

Figures 15 and 16 serve to illustrate the graphic technique as applied to sample Ferrographic data. Discussions based on these trial data plots from NAEC, MTI, and Foxboro are as follows:

- Figure 15 represents data from MTU/Mineral/Light; Transmitted/New Method. The " ΔM" of the NAEC measurement is approximately 2.3 times greater than the " ΔM" of Foxboro. The poor precision of the NAEC data, however, leads to no discrimination ability.

- Figure 16 represents data from MTU/Mineral/Heavy; Transmitted/New Method. The millimeter (mm) positions from 15 to 50 have a " ΔP" to " ΔM" ratio of 1.8 to 0.6; or approximately 3/1. The desired result for a reasonable ability to measure " ΔP" would have to be much greater, from about 6/1 to 10/1. As indicated by the line parallel to the X axis, Foxboro measurements are not correlated to the NAEC measurements above values of (25) because of a possible loss in discrimination capability. The results here are better than Figure 15, but are still considered inadequate.

- The Foxboro data indicates higher levels than NAEC by four points on the density scale, thus measuring the bias.

- For the positions from 0-10 mm, again the " ΔM" for the NAEC data is approximately 2.3 times greater than the " ΔM" of Foxboro. These are not considered useful measurements for heavy oil at these positions.

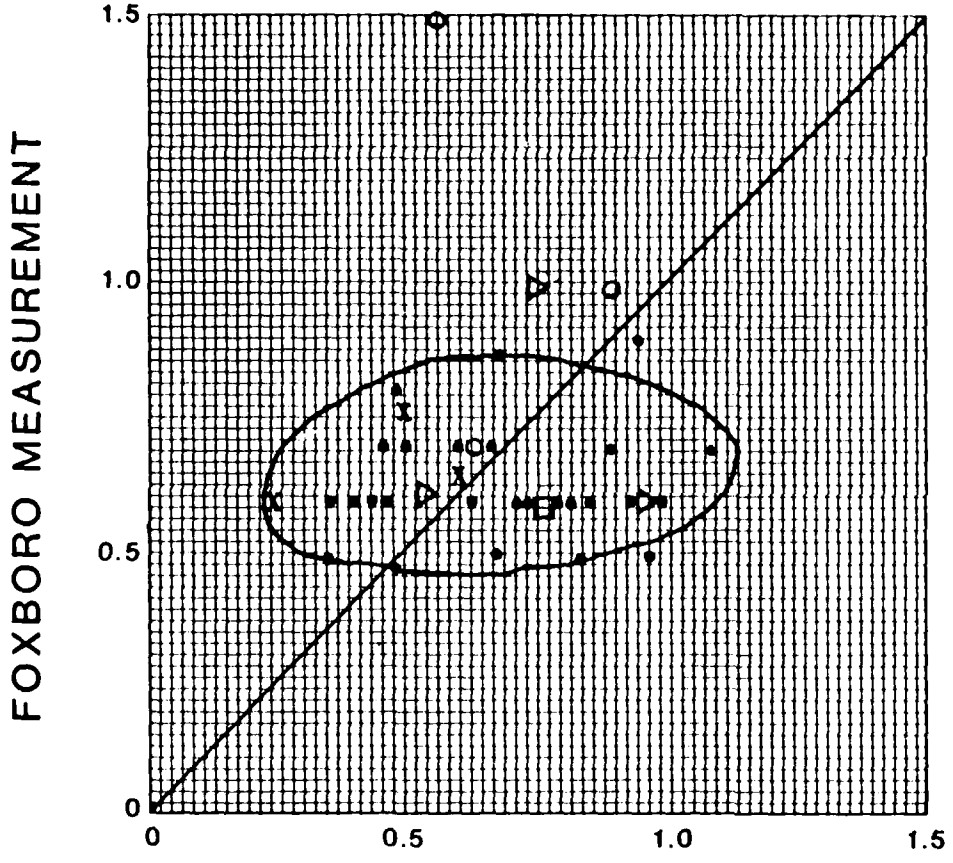
3.3.4 Statistical Analysis Summary

In order to summarize the above statistical discussion, outputs of the multifaceted statistical approach are listed as follows:

- Data Plots
  Trending Analysis
  Scatter Comparison
  Quantitative Comparisons

FIGURE 15

GRAPHICAL EVALUATION



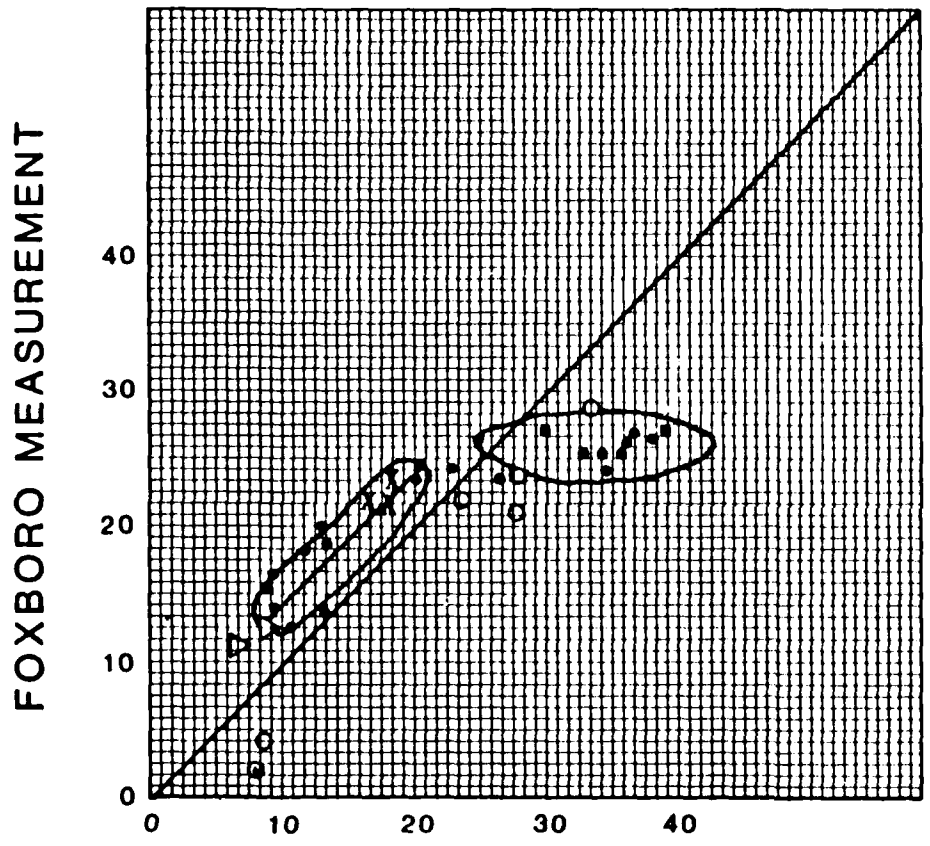
Position Legend, (mm)

- o 0mm
- x 20mm
- ▷ 50mm
- • All Others
- □ Duplicate Reading

MICHIGAN TECHNICAL UNIVERSITY/ MINERAL/LIGHT/ X-MITTED/NEW METHOD

FIGURE 16

GRAPHICAL EVALUATION



Position Legend, (mm)

- o 0mm
- x 20mm
- ▷ 50mm
- • All Others
- □ Duplicate Reading

MICHIGAN TECHNICAL UNIVERSITY/ MINERAL/HEAVY/ X-MITTED/NEW METHOD

- Analysis of Variance
  - Variable Significants
- Coefficient of Variation Analysis
  - Repeatability Assessment
  - Repeatability Apportionment
- Graphical Regression Analysis
  - Bias
  - Discrimination
  - Quantitative Comparisons
  - Scatter
  - Trending Analysis

4.0 PROGRAM RESULTS

Verification program results will be presented with respect to phase and stage. Due to the magnitude of the data generated under this program, only representative analysis examples will be cited where necessary. A complete volume of program data and analysis is on file both at the Office of Naval Research and Mechanical Technology Incorporated.

4.1 Phase I - Stage I

Results from the initial verification program stage are presented in the following summary. As described previously, this stage involved the analysis of fluid samples.

4.1.1 Data Plots

Representative Phase I - Stage I data plots are presented in Figures 17, 18, and 19.

The following results can be drawn from the analysis of the total plot population.

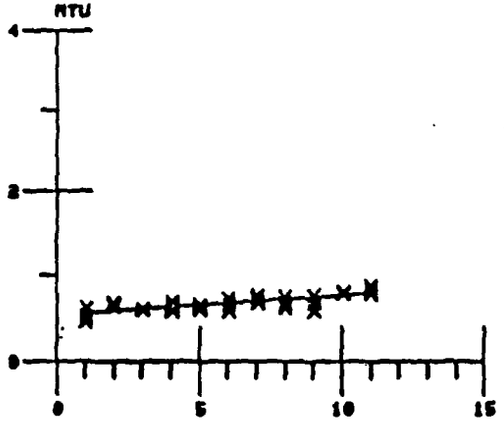
A. Trending agrees.

B. Quantitative variations exist.

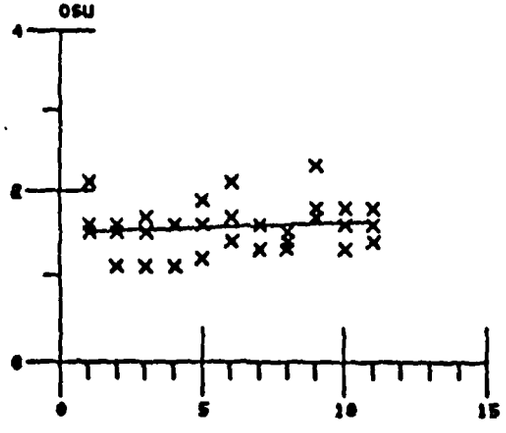
C. Lighting Technique as presented in Figure 20
   1) Mean of reflected higher than transmitted,
   2) Trends and standard deviation very similar.

D. Indexing Technique as presented in Figure 21
   1) Mean and standard deviation very similar.

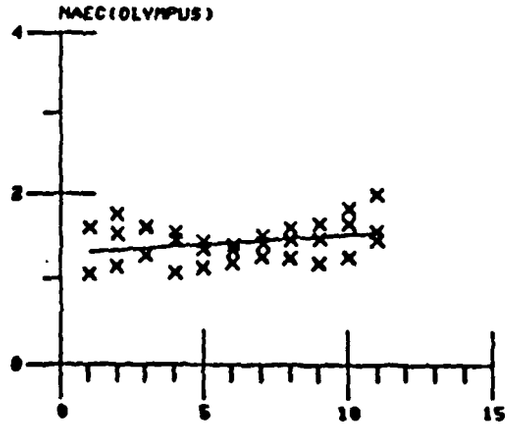
FIGURE 17



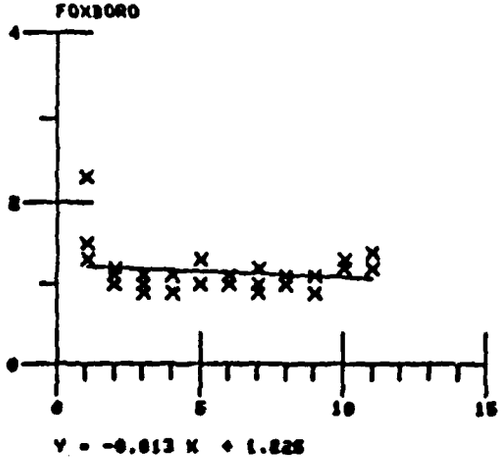
Y = 0.022 X + 0.540
YBAR = 0.674 SY = 0.094
SYX = 0.061 R = 0.757



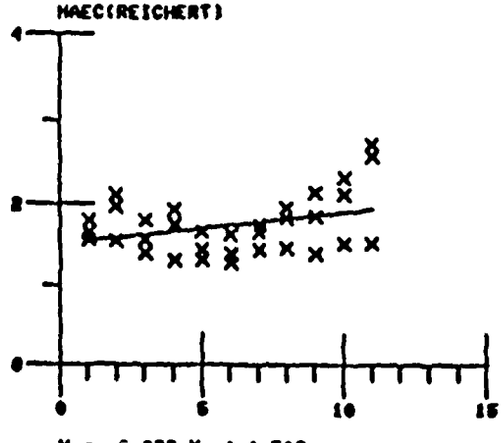
Y = 0.012 X + 1.498
YBAR = 1.573 SY = 0.082
SYX = 0.284 R = 0.000



Y = 0.024 X + 1.283
YBAR = 1.426 SY = 0.231
SYX = 0.221 R = 0.285



Y = -0.013 X + 1.226
YBAR = 1.145 SY = 0.258
SYX = 0.256 R = 0.000



Y = 0.038 X + 1.582
YBAR = 1.731 SY = 0.353
SYX = 0.336 R = 0.305

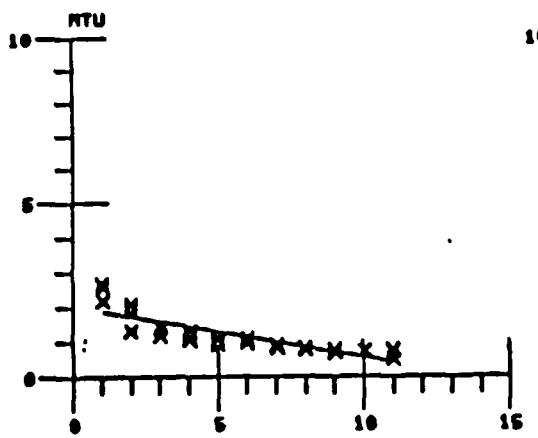
PHASE I STAGE I NTI PH087

FERROGRAPHY DATA

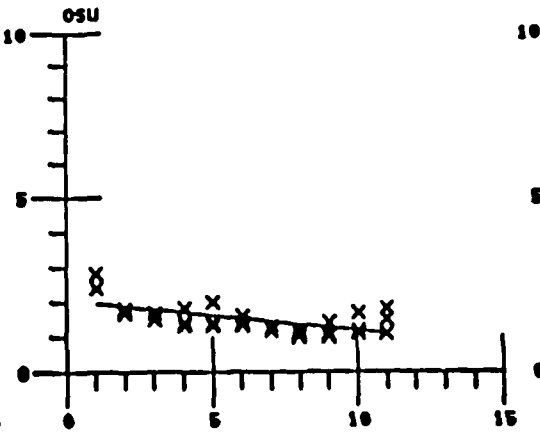
DENSITY VS SLIDE POSITION

NTU MINERAL
LIGHT
REFLECTED
NEW METHOD

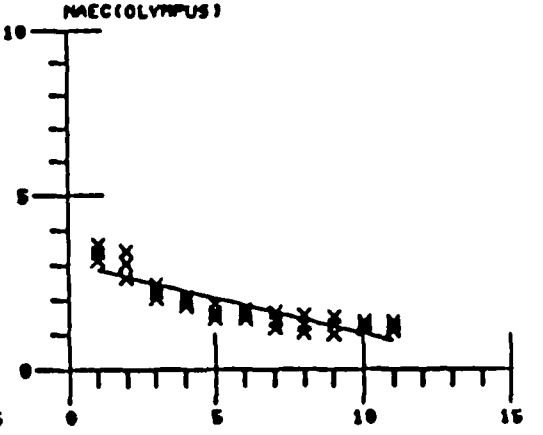
FIGURE 18



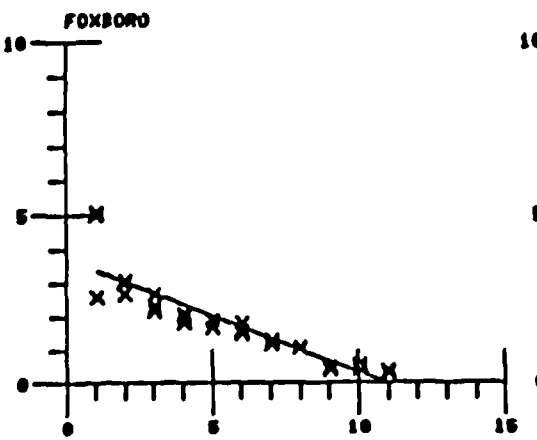
Y = -0.146 X + 8.021
YBAR = 1.145 SY = 0.556
SYX = 0.382 R = 0.839



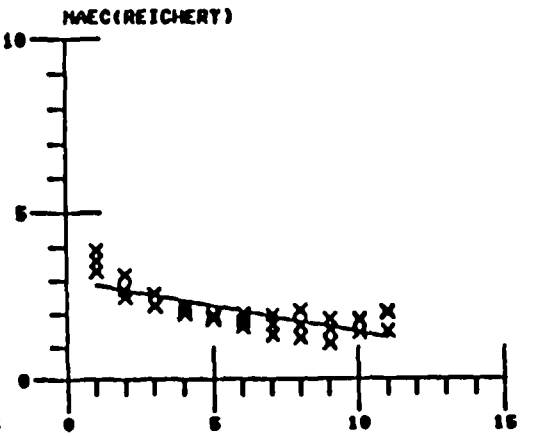
Y = -0.085 X + 8.028
YBAR = 1.515 SY = 0.424
SYX = 0.328 R = 0.633



Y = -0.203 X + 3.058
YBAR = 1.842 SY = 0.745
SYX = 0.368 R = 0.869



Y = -0.338 X + 3.718
YBAR = 1.688 SY = 1.200
SYX = 0.618 R = 0.862



Y = -0.155 X + 3.000
YBAR = 2.069 SY = 0.654
SYX = 0.430 R = 0.753

PHASE I STAGE I MTI PH887

FERROGRAPHY DATA

DENSITY VS SLIDE POSITION

NAEC SYNTHETIC
MEDIUM
REFLECTED
CONVENTIONAL

FIGURE 19

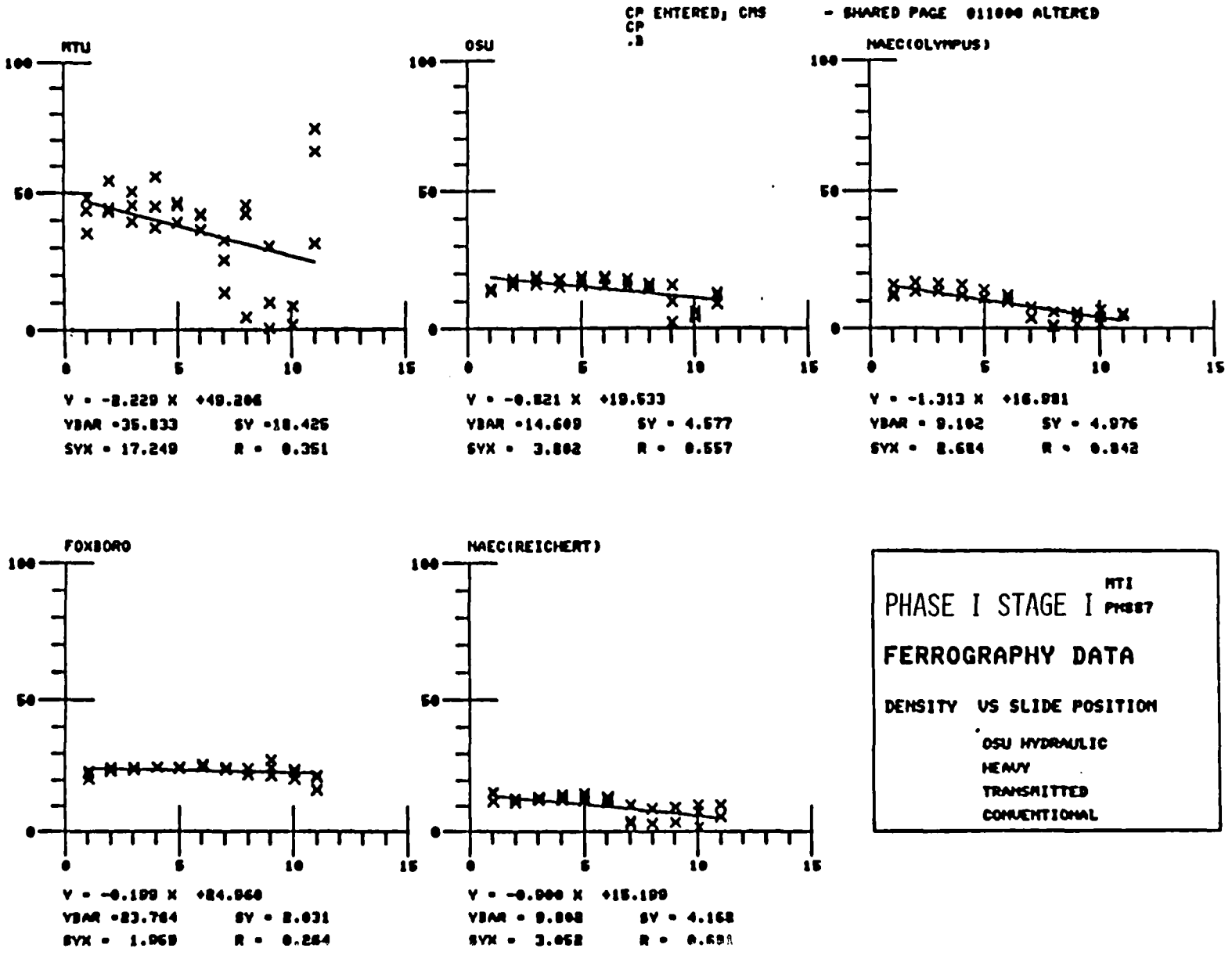

CP ENTERED; CMS - SHARED PAGE 011000 ALTERED
CP
.B
MTU
100
50
0
0
5
10
15
Y = -8.229 X +49.206
YBAR =35.833 SY =18.425
SYX = 17.249 R = 0.351
OSU
100
50
0
0
5
10
15
Y = -0.821 X +19.533
YBAR =14.609 SY = 4.577
SYX = 3.882 R = 0.557
HAEC(OLYMPUS)
100
50
0
0
5
10
15
Y = -1.313 X +16.981
YBAR = 9.102 SY = 4.976
SYX = 8.684 R = 0.842
FOXBORO
100
50
0
0
5
10
15
Y = -0.199 X +24.960
YBAR =23.764 SY = 2.031
SYX = 1.969 R = 0.284
HAEC(REICHERT)
100
50
0
0
5
10
15
Y = -0.900 X +15.199
YBAR = 9.868 SY = 4.168
SYX = 3.052 R = 0.691
PHASE I STAGE I
MTI
PH887
FERROGRAPHY DATA
DENSITY VS SLIDE POSITION
OSU HYDRAULIC
HEAVY
TRANSMITTED
CONVENTIONAL

FIGURE 20

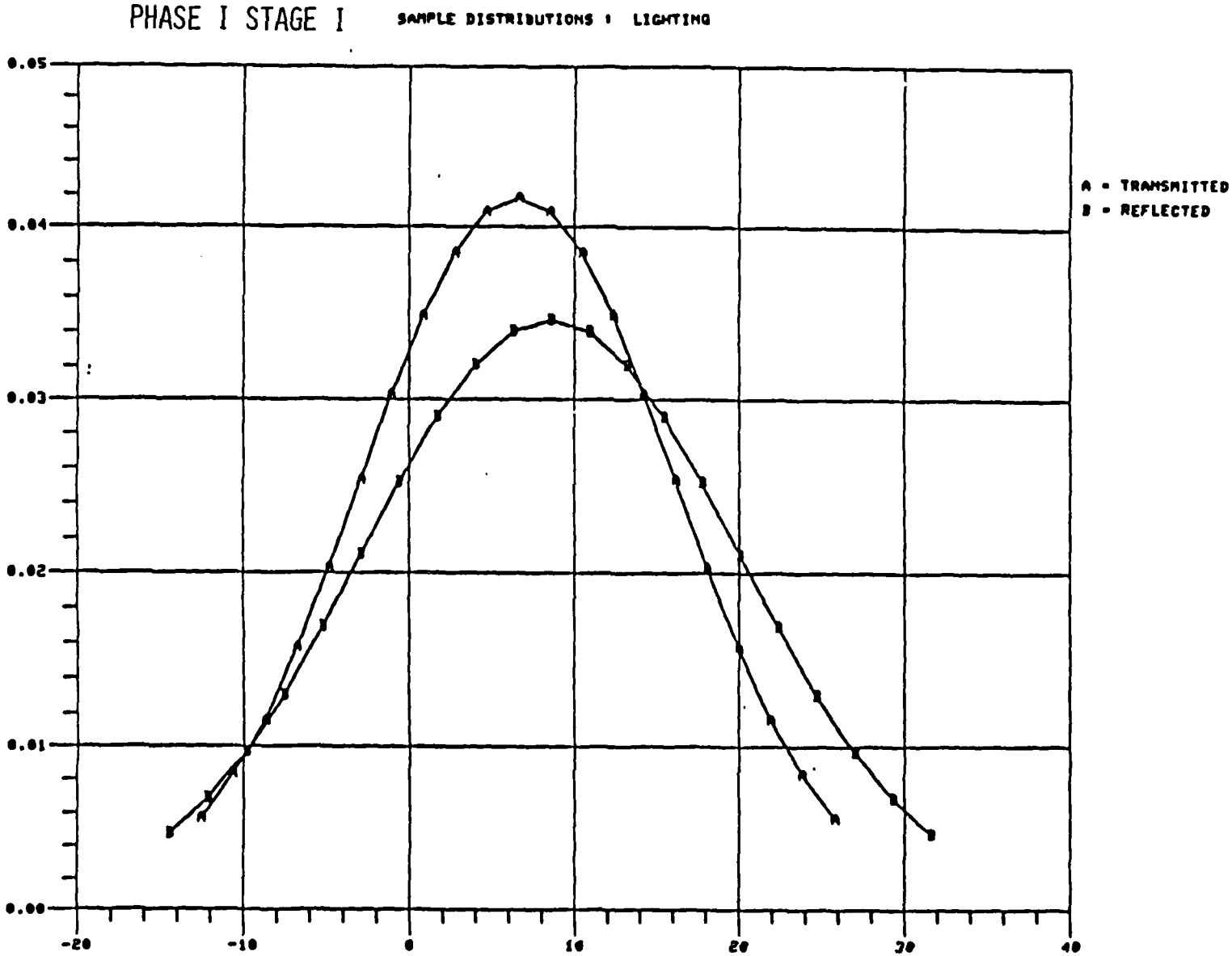
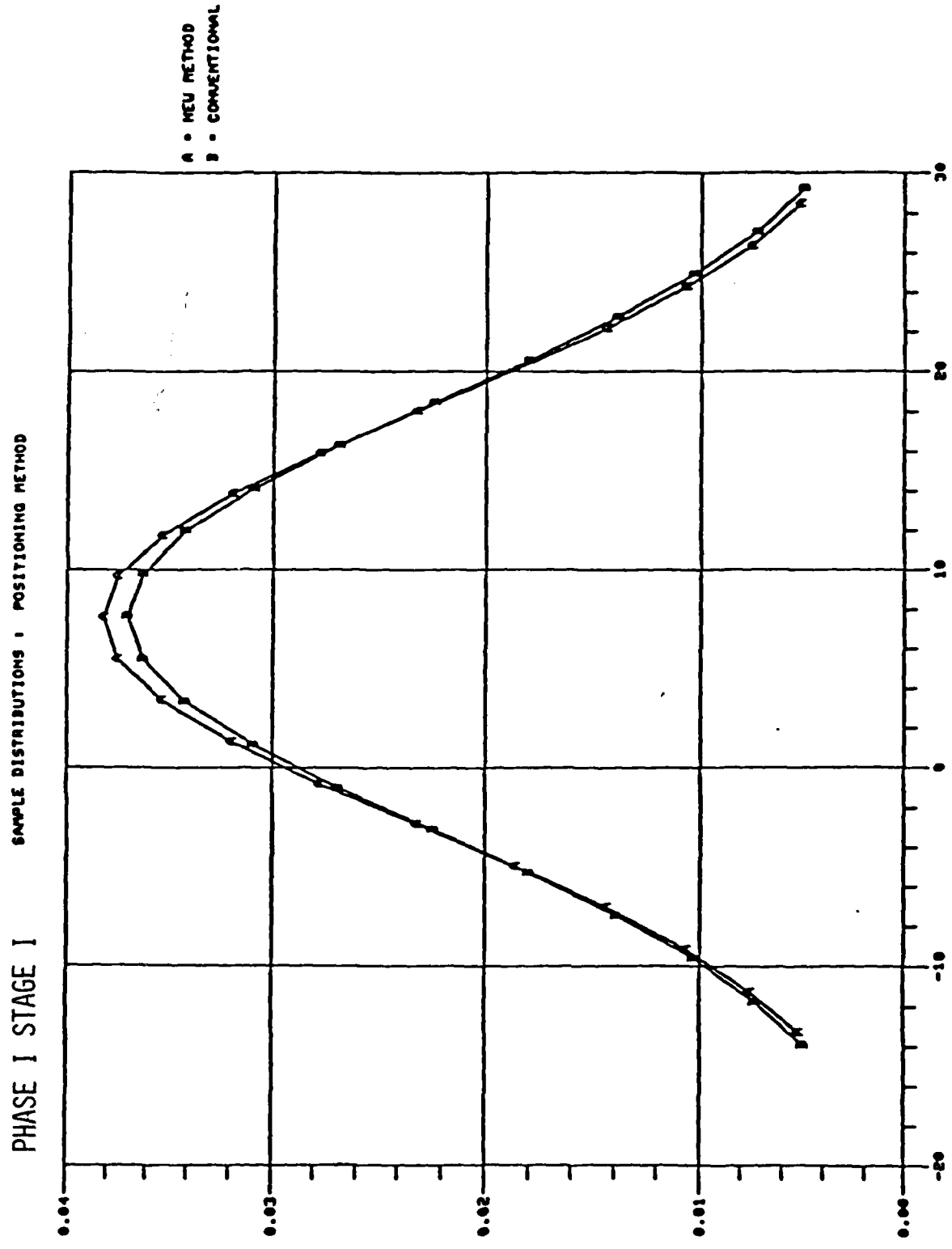

PHASE I STAGE I
SAMPLE DISTRIBUTIONS : LIGHTING
0.05
0.04
0.03
0.02
0.01
0.00
-20
-10
0
10
20
30
40
A = TRANSMITTED
B = REFLECTED

FIGURE 21


PHASE I STAGE I
SAMPLE DISTRIBUTIONS - POSITIONING METHOD
A = NEW METHOD
B = CONVENTIONAL
0.04
0.03
0.02
0.01
0.00
30
20
10
0
-10
-20

E. Sample Type and Concentration as presented in Figure 22

1) Synthetic samples are suspect:

(a) Low mean concentration,

(b) Tight distribution,

(c) Similar light and medium concentration levels,

(d) Light "heavy sample" concentration.

2) Hydraulic and mineral samples very similar.

F. Equipment

1) Reichert and Olympus microscope mean and standard deviation very similar

	REC	OLY
- Mean Density	7.9	7.24
- Standard Deviation	11.51	11.03

G. Slide Position

1) Observations consistent over slide length/slide position.

H. Laboratories as presented in Figure 23

1) Mean density value of MTU, NAEC (OLY), NAEC (REC), and Foxboro similar as

- Mean Value $\simeq$ 8.0

2) Standard deviation for MTU, NAEC (OLY), and NAEC (REC) similar

- Standard Deviation $\simeq$13.0

3) OSU mean density relatively low

- OSU Mean Value $\simeq$ 5.6

4) OSU and Foxboro standard deviation relatively low

- OSU Std. Dev. $\simeq$ 6.4
- Foxboro Std. Dev. $\simeq$ 9.2

5) OSU and Foxboro deviated from dilution procedure

4.1.2 Analysis of Variation as presented in Table 1

A. Type of oil is a significant variable.

B. Debris Concentration is a significant variable.

C. Laboratory is a significant variable.

FIGURE 22

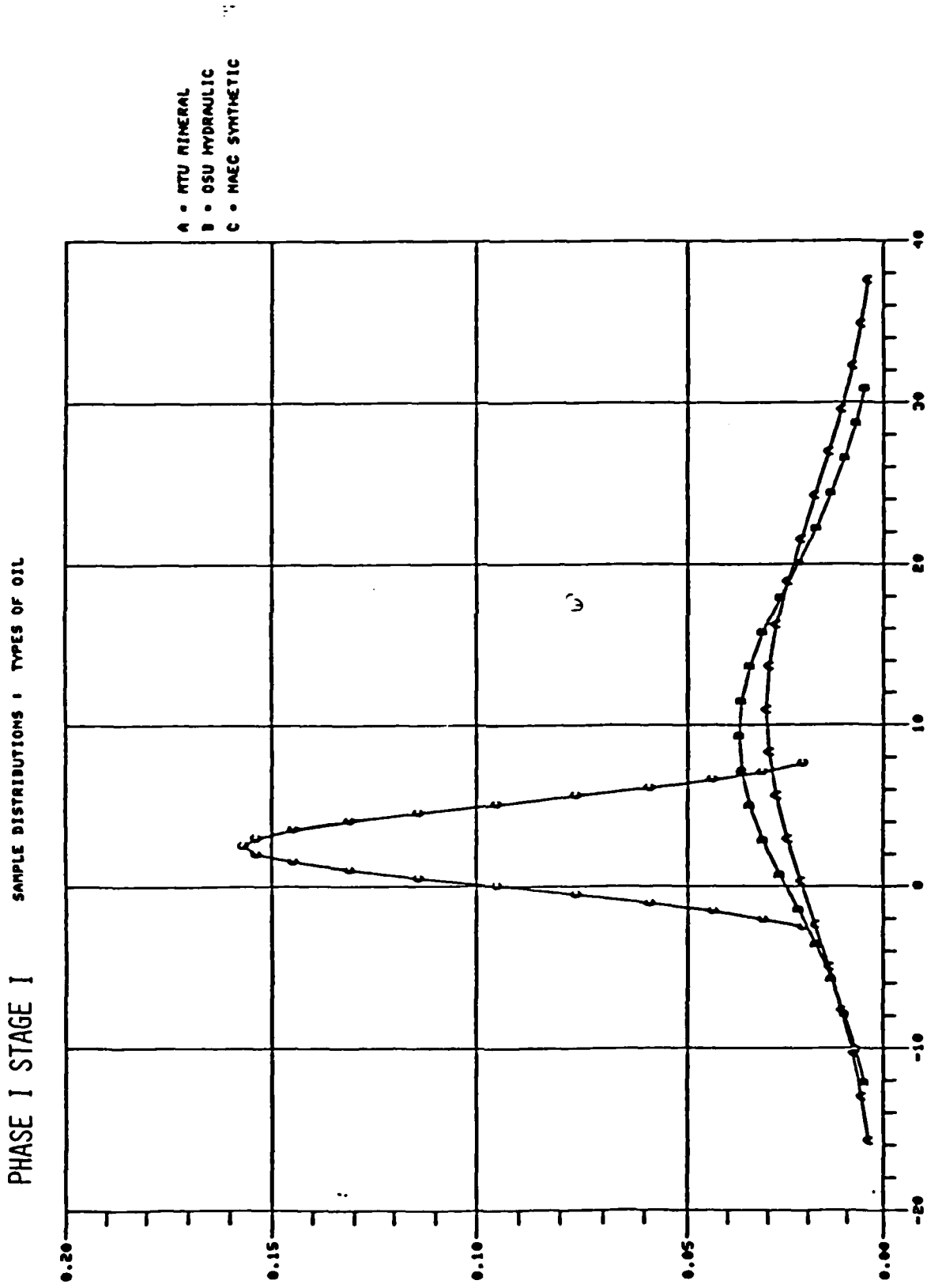

PHASE I STAGE I
SAMPLE DISTRIBUTIONS : TYPES OF OIL
A = MTU MINERAL
B = OSU HYDRAULIC
C = NAEC SYNTHETIC
0.20
0.15
0.10
0.05
0.00
40
30
20
10
0
-10
-20

FIGURE 23

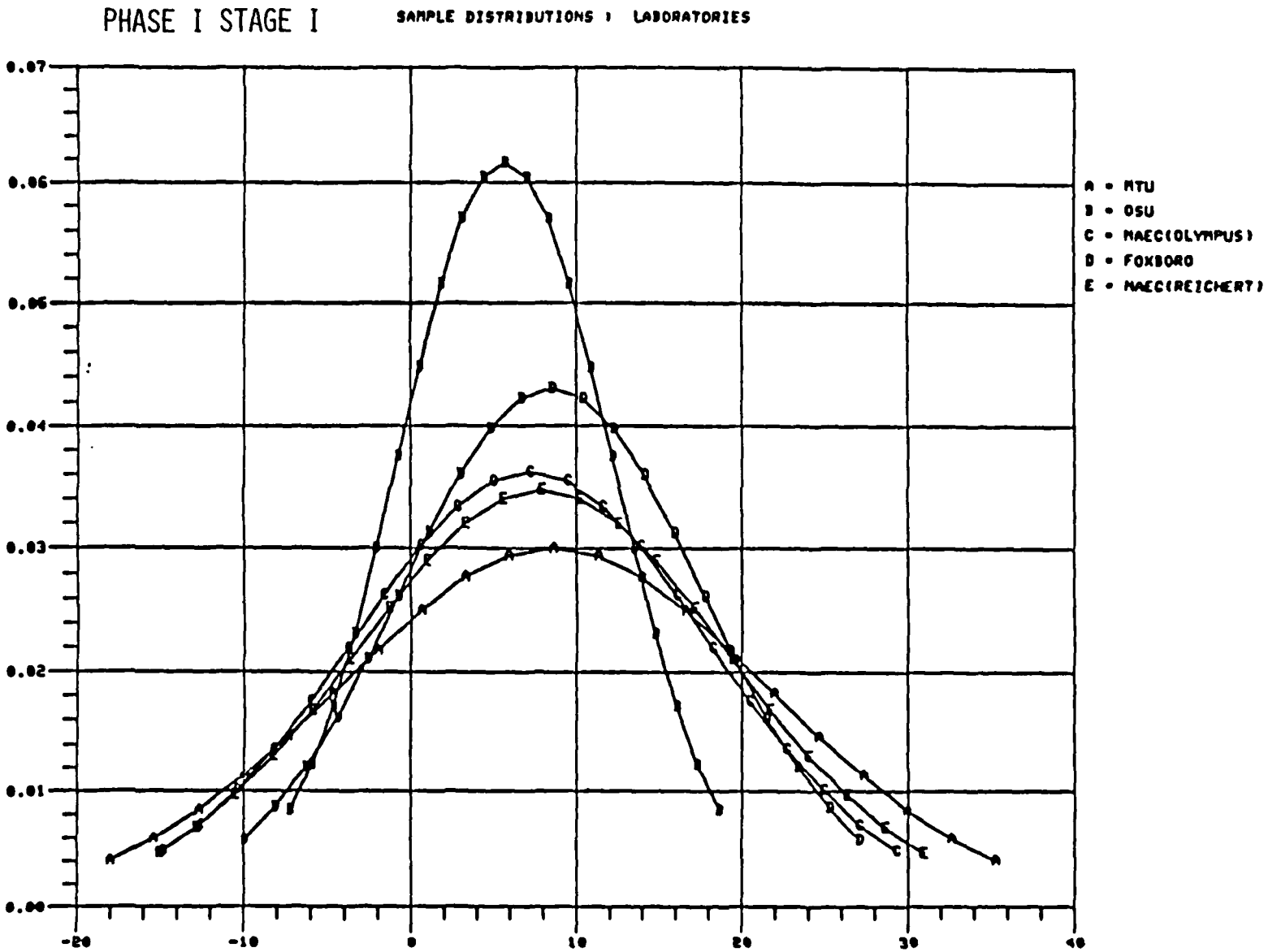

PHASE I STAGE I
SAMPLE DISTRIBUTIONS : LABORATORIES
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
-20
-10
0
10
20
30
40
A = MTU
B = OSU
C = MAEC(OLYMPUS)
D = FOXBORO
E = MAEC(REICHERT)

TABLE 1

PHASE I STAGE I

ANALYSIS OF VARIANCE FERROGRAPHY DATA

SOURCE OF VARIATION	SUM OF SQUARES SS	DEGREES OF FREEDOM DF	MEAN SQUARE MS/DF	MEAN-SQUARE RATIO MSR MS/MSRESID	F VALUE 90 PERCENT CONFIDENCE	CONCLUSION
TYPE OF OIL	0.7903E+05	2.	0.3952E+05	0.7807E+04	2.30	SIGNIFICANTLY EFFECTS DENSITY
CONCENTRATION	0.2713E+06	2.	0.1357E+06	0.2680E+05	2.30	SIGNIFICANTLY EFFECTS DENSITY
LABORATORY	0.6857E+04	4.	0.1714E+04	0.3387E+03	1.94	SIGNIFICANTLY EFFECTS DENSITY
LIGHTING	0.5794E+04	1.	0.5794E+04	0.1145E+04	2.71	SIGNIFICANTLY EFFECTS DENSITY
METHOD	0.9791E+01	1.	0.9791E+01	0.1934E+01	2.71	EFFECT NOT SIGNIFICANT
SLIDE POSITION	0.2169E+05	10.	0.2169E+04	0.4286E+03	1.60	SIGNIFICANTLY EFFECTS DENSITY

D. Lighting Technique is a significant variable.

E. Index Technique is not a significant variable.

F. Slide position is a significant variable.

4.1.3 Coefficient of Variation

A. Intra-Laboratory as presented in Figure 24

1) Range $\simeq$ 0 - 42%

Mean $\simeq$ 19%

2) Sample Type

(a) Slight variation between types of fluid

- Mean COV Mineral $\simeq$ 15%
- Mean COV Hydraulic $\simeq$ 21%
- Mean COV Synthetic $\simeq$ 17%

3) Sample Concentration as presented in Figure 25

(a) Light and heavy concentrations most significant

- Mean COV Light $\simeq$ 22%
- Mean COV Medium $\simeq$ 13%
- Mean COV Heavy $\simeq$ 19%

4) Lighting Technique as presented in Figure 26

(a) Very similar COV between lighting techniques.

5) Indexing Technique as presented in Figure 27

(a) Very similar COV between indexing techniques.

6) Equipment

(a) Very similar COV between microscopes.

7) Slide Position

(a) COV consistent with respect to slide position

8) Laboratory as presented in Figure 28

(a) MTU, OSU, Foxboro, and NAEC (OLY) similar

- Mean COV $\simeq$ 17%

(b) NAEC (REC)

- Mean COV $\simeq$ 14%

B. Interlaboratory as presented in Table 2

1) Range $\simeq$ 10 - 74%

Mean $\simeq$ 50%

2) Sample Type

(a) Slight variation between types of fluid.

FIGURE 24

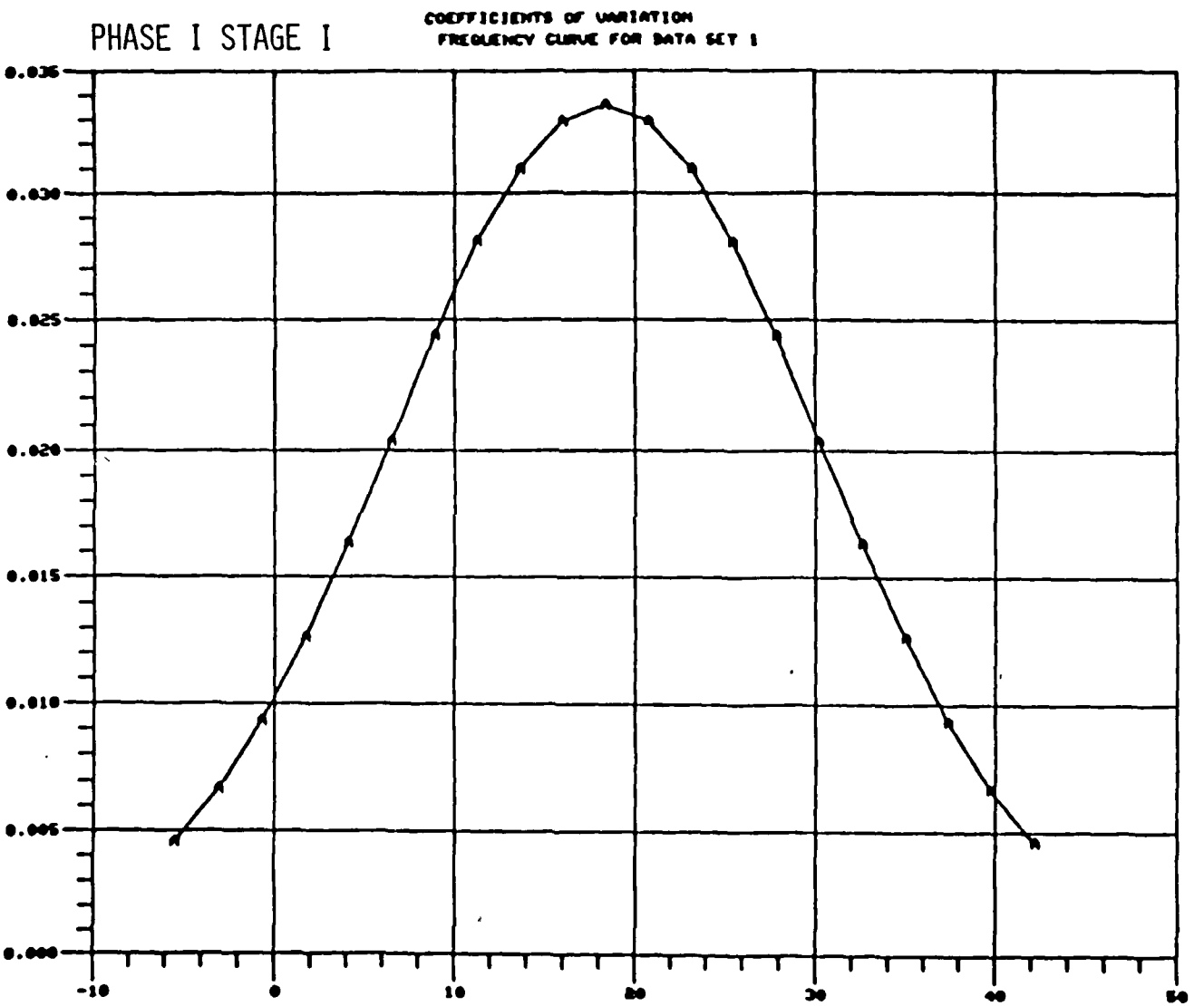

PHASE I STAGE I
COEFFICIENTS OF VARIATION
FREQUENCY CURVE FOR DATA SET 1
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-10
0
10
20
30
40
50

FIGURE 25

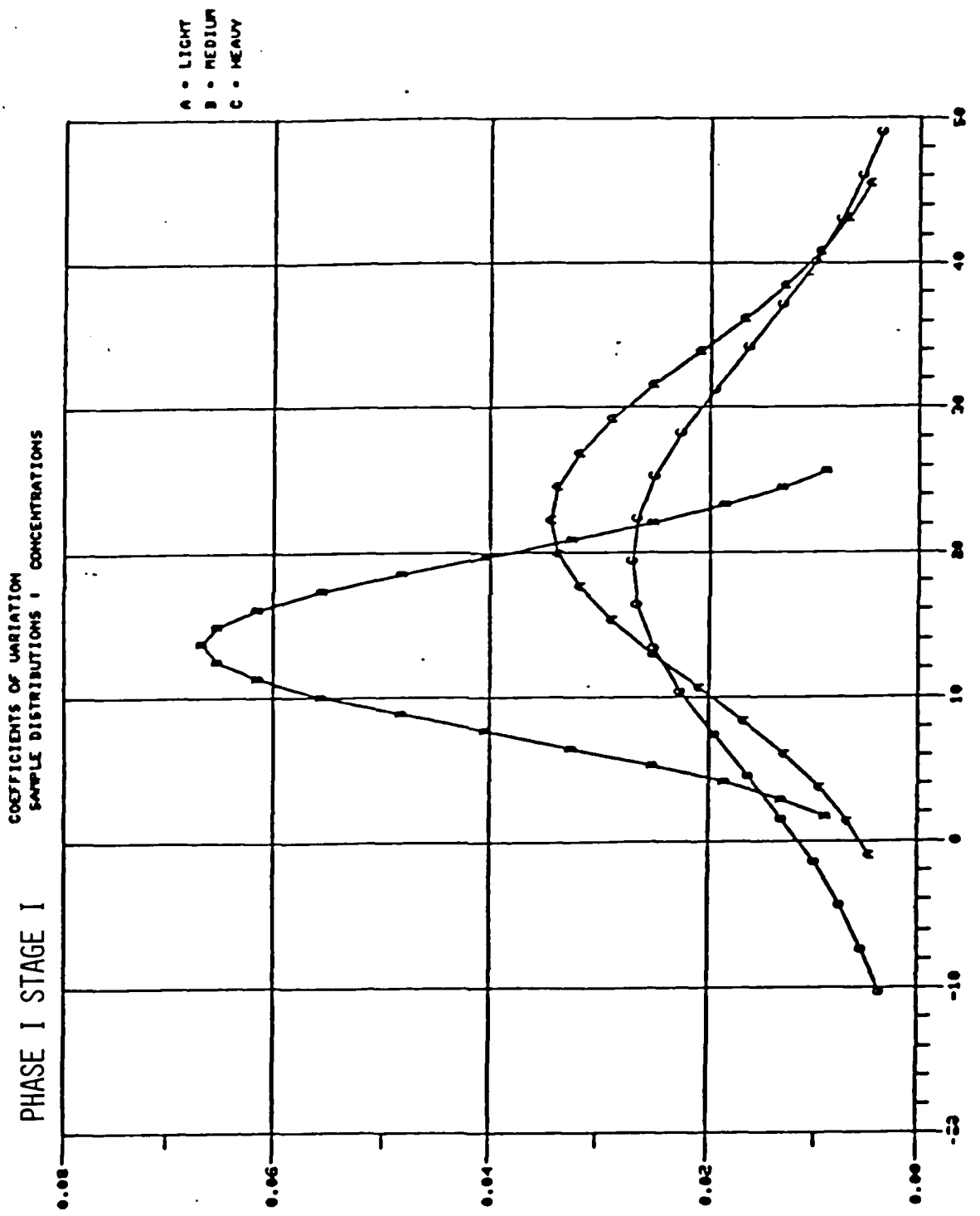

PHASE I STAGE I
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS : CONCENTRATIONS
A = LIGHT
B = MEDIUM
C = HEAVY
0.08
0.06
0.04
0.02
0.00
-20
-10
0
10
20
40
50

FIGURE 26

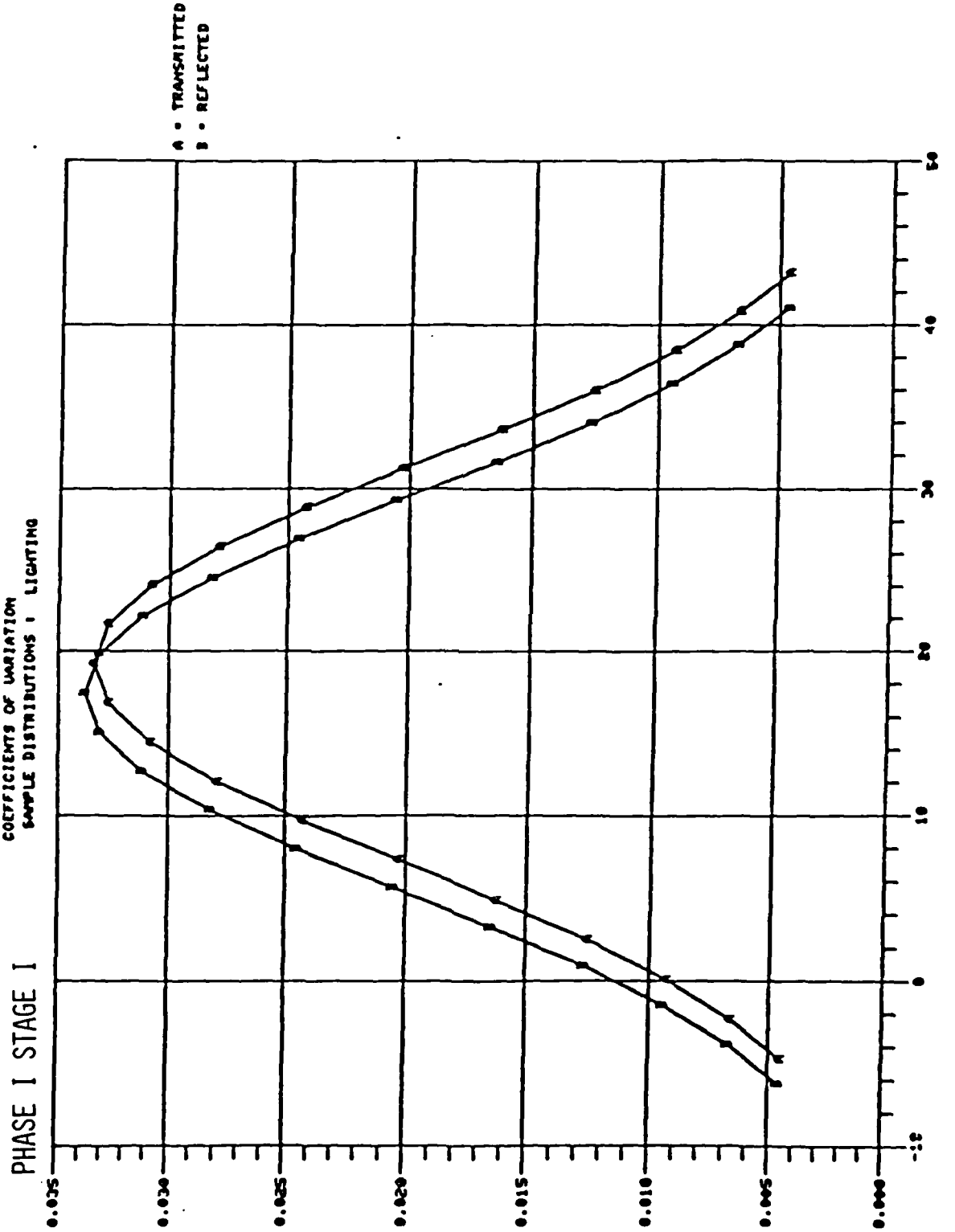

A = TRANSMITTED
B = REFLECTED
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS : LIGHTING
PHASE I STAGE I
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-10
0
10
20
30
40
50

FIGURE 27

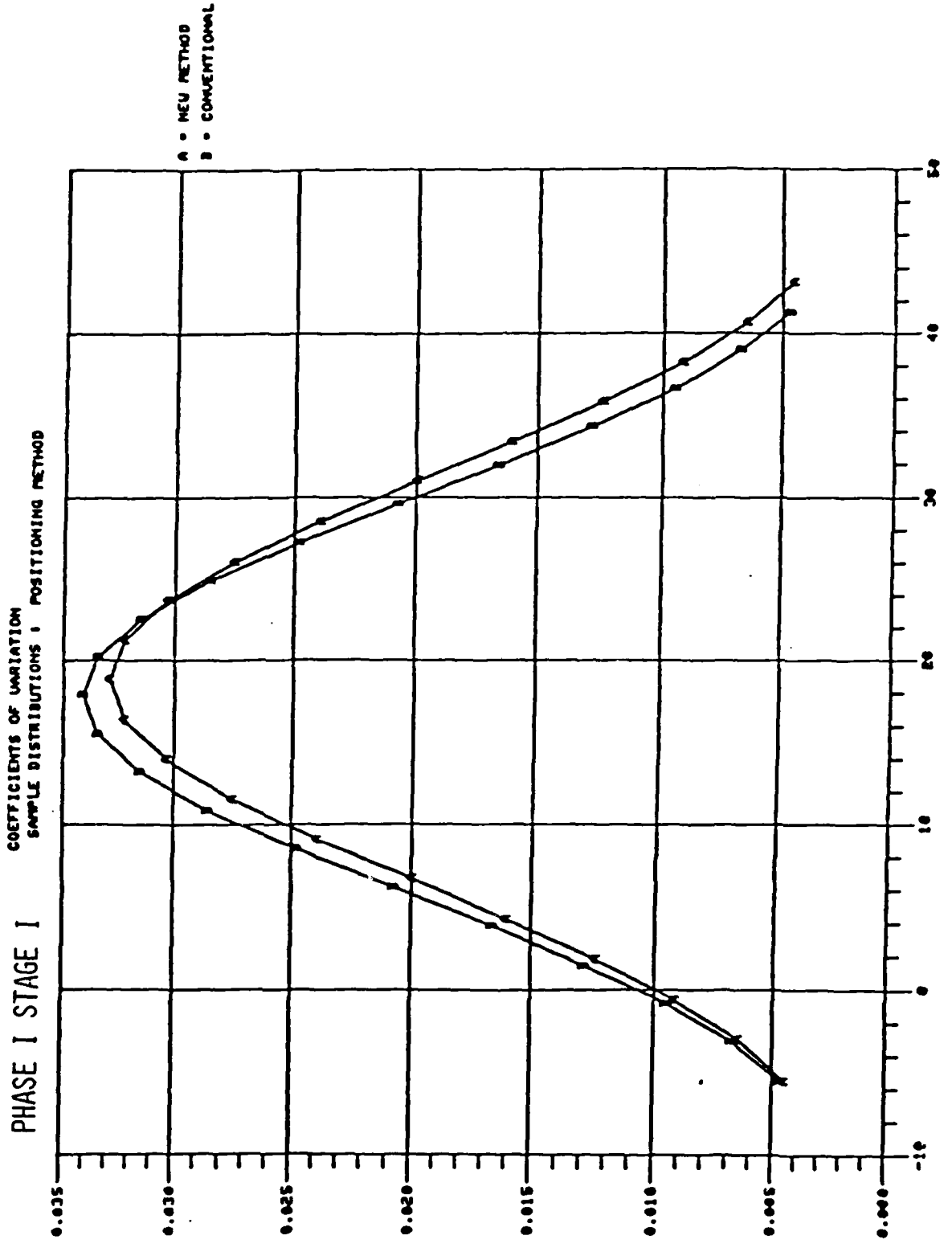

A = NEW METHOD
B = CONVENTIONAL
PHASE I STAGE I
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS : POSITIONING METHOD
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-10
0
10
20
30
40
50

FIGURE 28

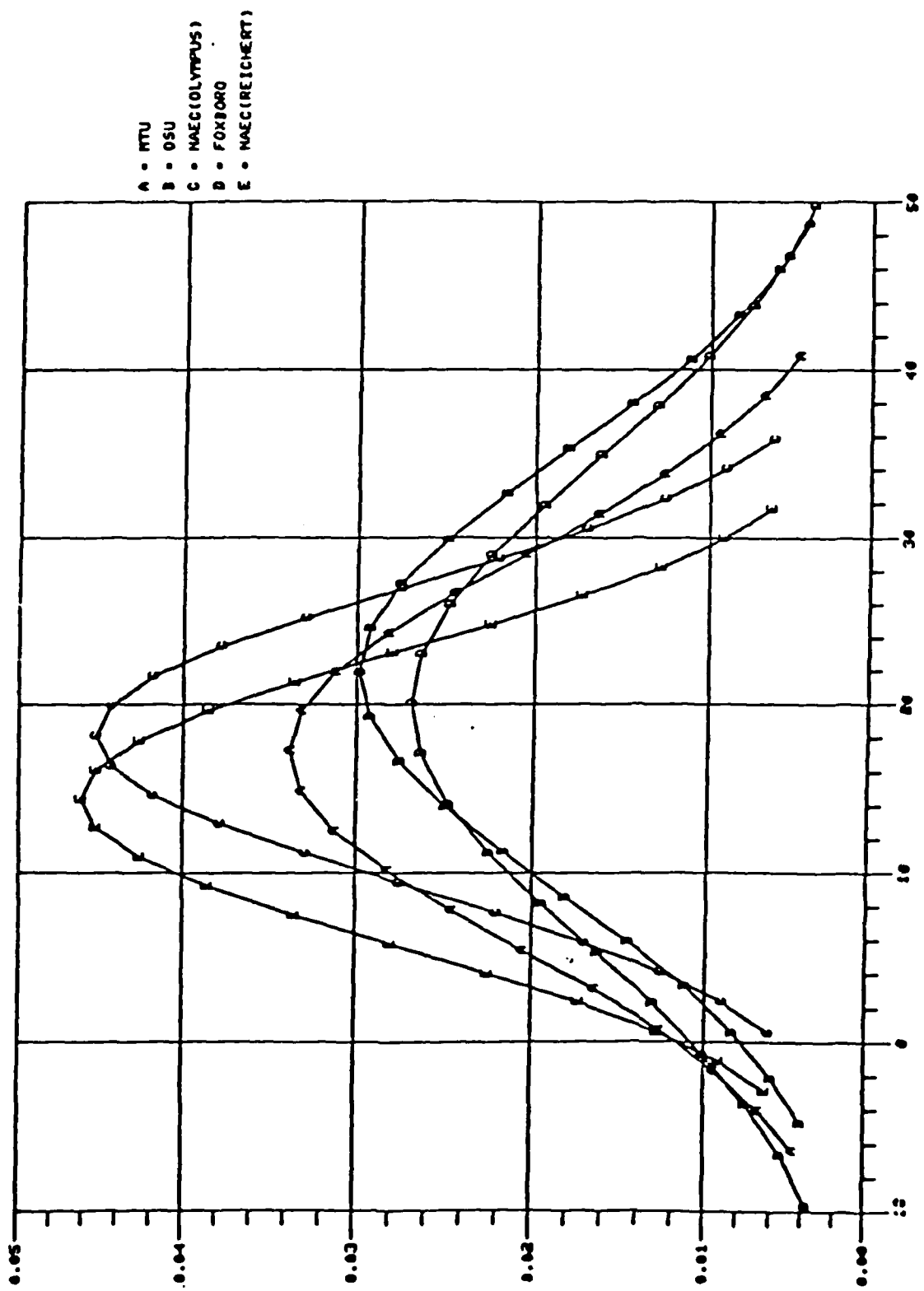

PHASE I STAGE I
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS - LABORATORIES
A = RTU
B = OSU
C = NAEC(OLYMPUS)
D = FOXBORO
E = NAEC(REICHERT)
0.05
0.04
0.03
0.02
0.01
0.00
10
0
10
20
30
40
50

TABLE 2

PHASE I STAGE I

COEFFICIENT OF VARIATION INTERLABORATORY

SLIDE POSITION	TYPE OF OIL	CONCENTRATION	COEFF. OF VARIATION
1	MTU MINERAL	LIGHT	0.4959E+02
5	MTU MINERAL	LIGHT	0.4109E+02
10	MTU MINERAL	LIGHT	0.4648E+02
1	MTU MINERAL	MEDIUM	0.3186E+02
5	MTU MINERAL	MEDIUM	0.3000E+02
10	MTU MINERAL	MEDIUM	0.3489E+02
1	MTU MINERAL	HEAVY	0.3460E+02
5	MTU MINERAL	HEAVY	0.3651E+02
10	MTU MINERAL	HEAVY	0.3777E+02
1	OSU HYDRAULIC	LIGHT	0.3652E+02
5	OSU HYDRAULIC	LIGHT	0.3885E+02
10	OSU HYDRAULIC	LIGHT	0.1043E+03
1	OSU HYDRAULIC	MEDIUM	0.2716E+02
5	OSU HYDRAULIC	MEDIUM	0.2119E+02
10	OSU HYDRAULIC	MEDIUM	0.3532E+02
1	OSU HYDRAULIC	HEAVY	0.5563E+02
5	OSU HYDRAULIC	HEAVY	0.5441E+02
10	OSU HYDRAULIC	HEAVY	0.7451E+02
1	NAEC SYNTHETIC	LIGHT	0.4704E+02
5	NAEC SYNTHETIC	LIGHT	0.4052E+02
10	NAEC SYNTHETIC	LIGHT	0.5512E+02
1	NAEC SYNTHETIC	MEDIUM	0.3426E+02
5	NAEC SYNTHETIC	MEDIUM	0.3109E+02
10	NAEC SYNTHETIC	MEDIUM	0.5122E+02
1	NAEC SYNTHETIC	HEAVY	0.6861E+02
5	NAEC SYNTHETIC	HEAVY	0.2936E+02
10	NAEC SYNTHETIC	HEAVY	0.5209E+02

3) Sample Concentration
   (a) Light and medium mean COV $\simeq$ 39%
   (b) Heavy mean COV $\simeq$ 57%
4) Slide Position
   (a) COV consistent over slide position

4.1.4 Graphical Regression Analysis

Representative Phase I - Stage I regression analysis plots are presented in Figures 29, 30, and 31.

The following results can be drawn from the analysis of the total regression analysis plot population.

A. Poor quantitative interlaboratory correlation exists.
B. Inconsistent data bias exists.
C. Poor discrimination as presented in Figure 32
   Mean $\simeq$ .91
D. Substantial scatter exists.

4.2 Phase I - Stage II

Results from the second verification program stage is presented in the following summary. As described previously, this stage involved the analysis of pre-made Ferrogram slides.

4.2.1 Data Plots

Representative Phase I - Stage II data plots are presented in Figures 33, 34, and 35.

The following results can be drawn from the analysis of the total plot population.

A. Trending agrees

B. Quantitative variations exist

FIGURE 29

PHASE I STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOTS

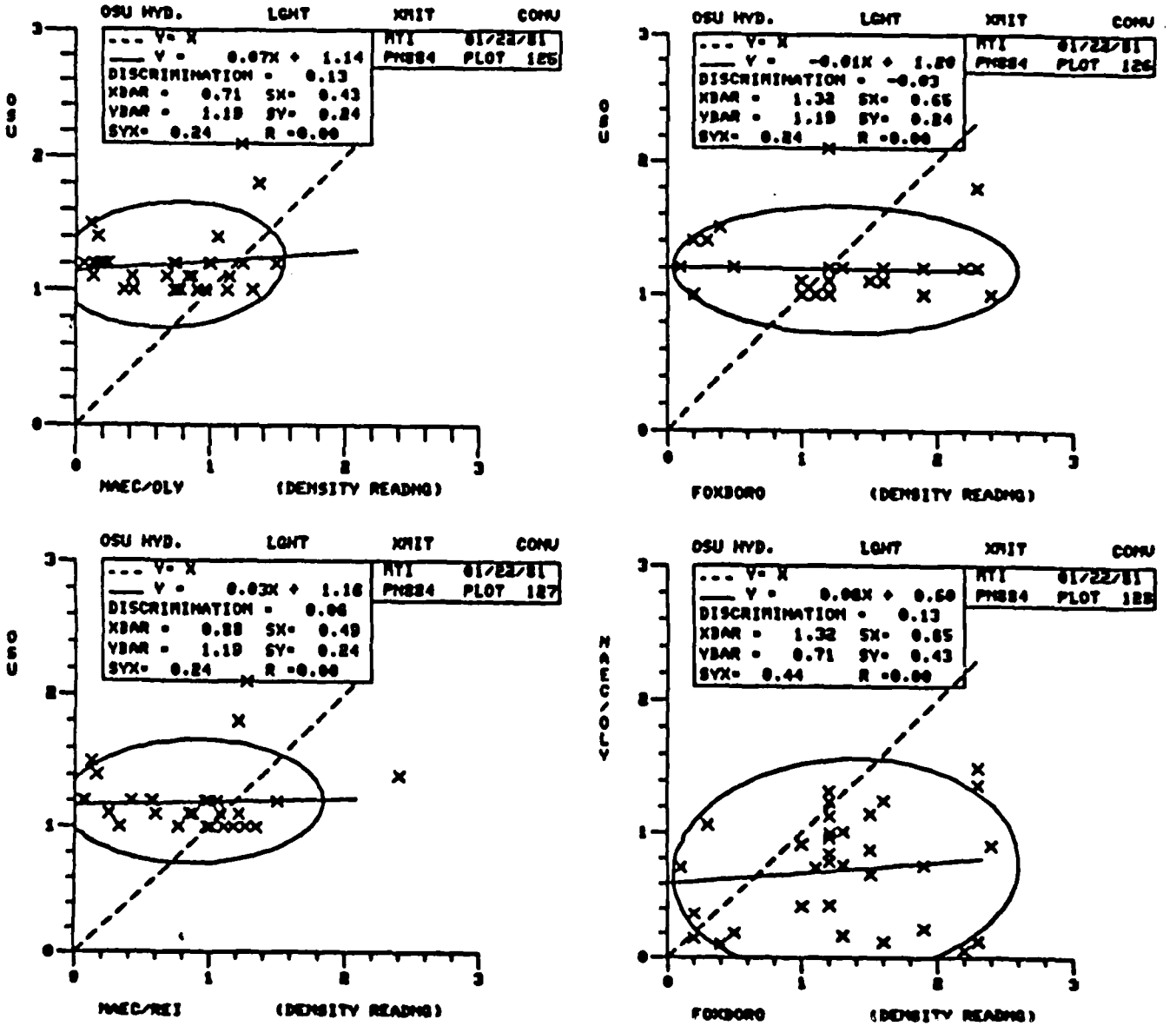


FIGURE 30

PHAGE I STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOTS

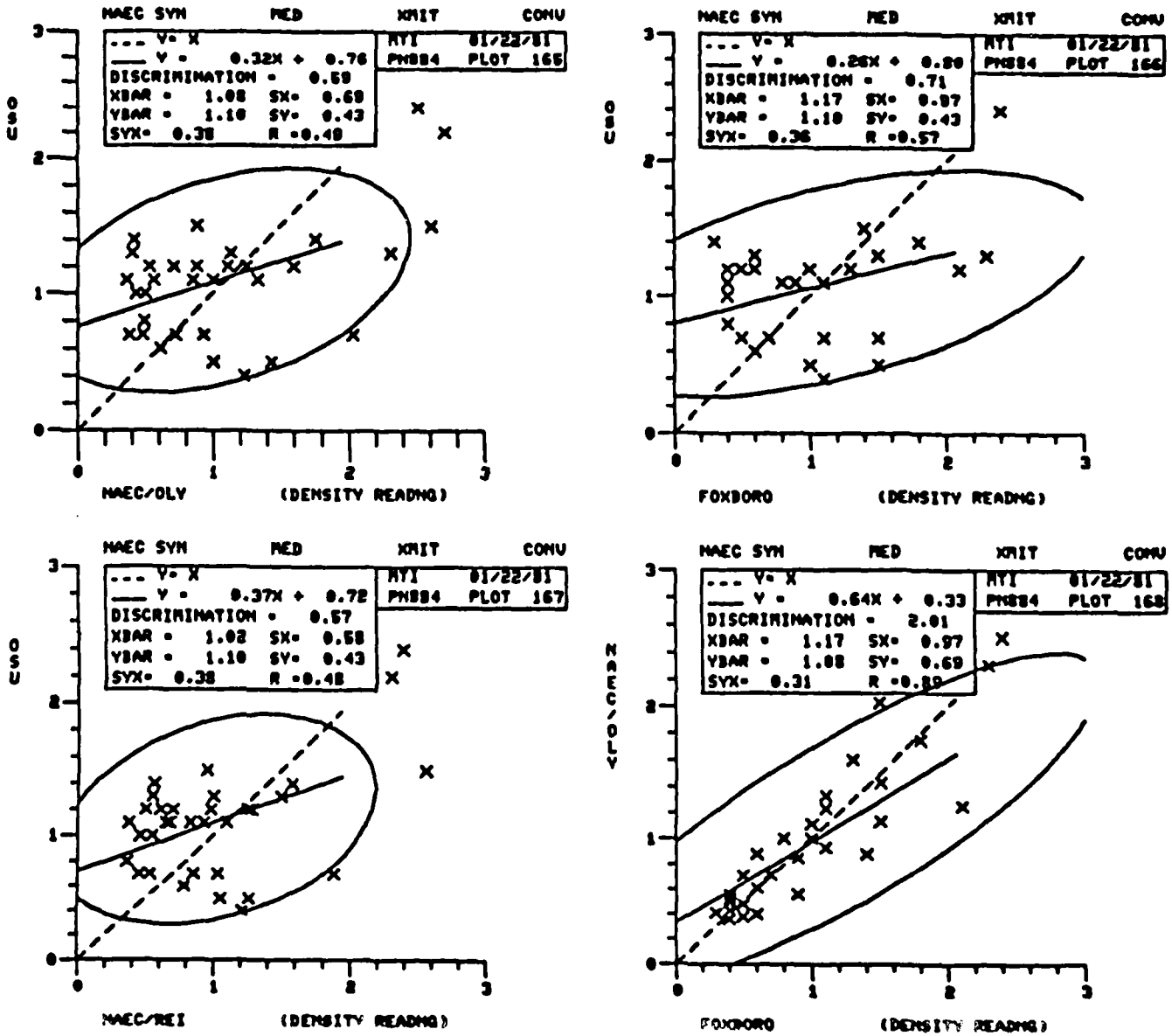


FIGURE 31

PHAGE I STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOTS

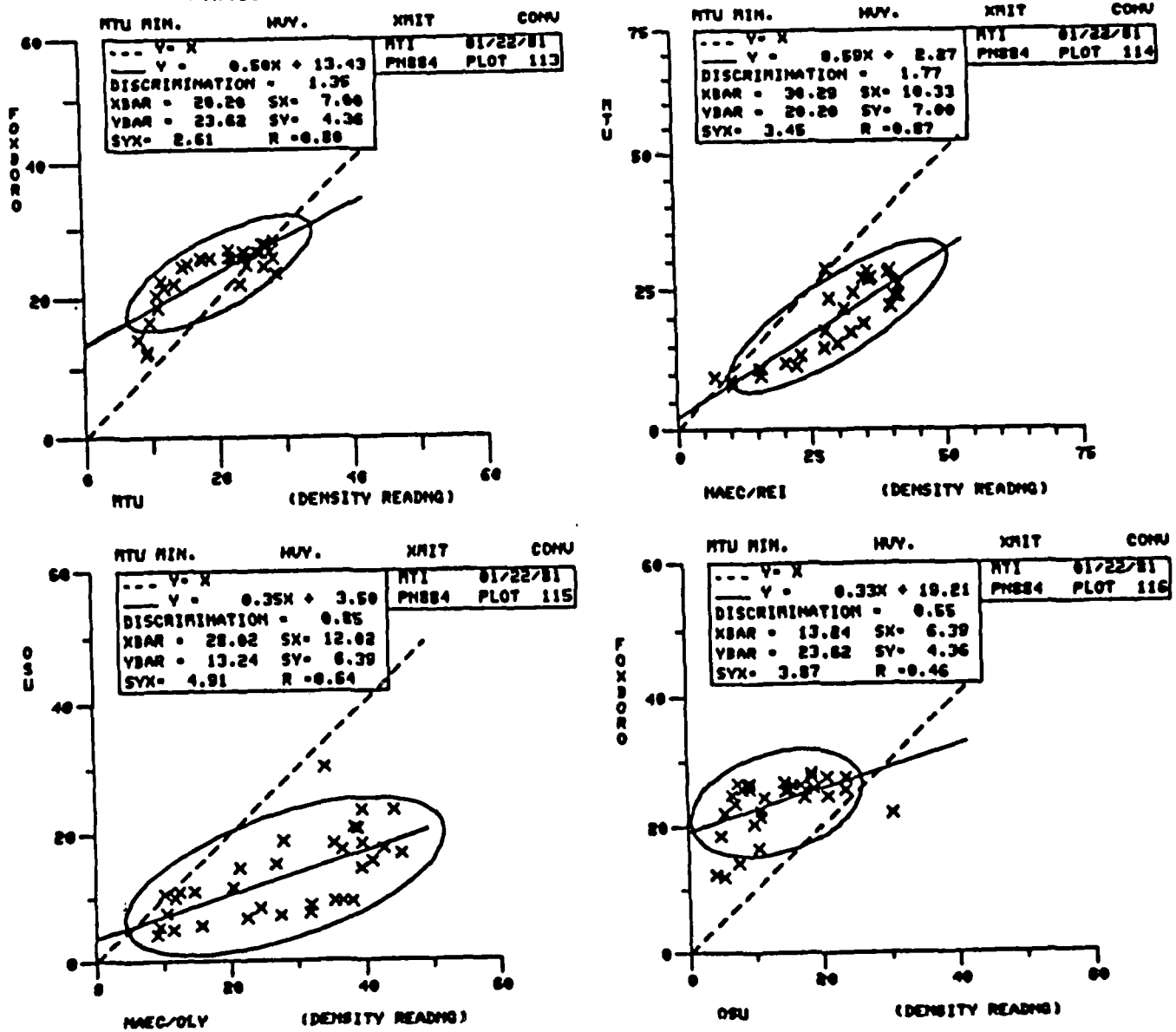


FIGURE 32

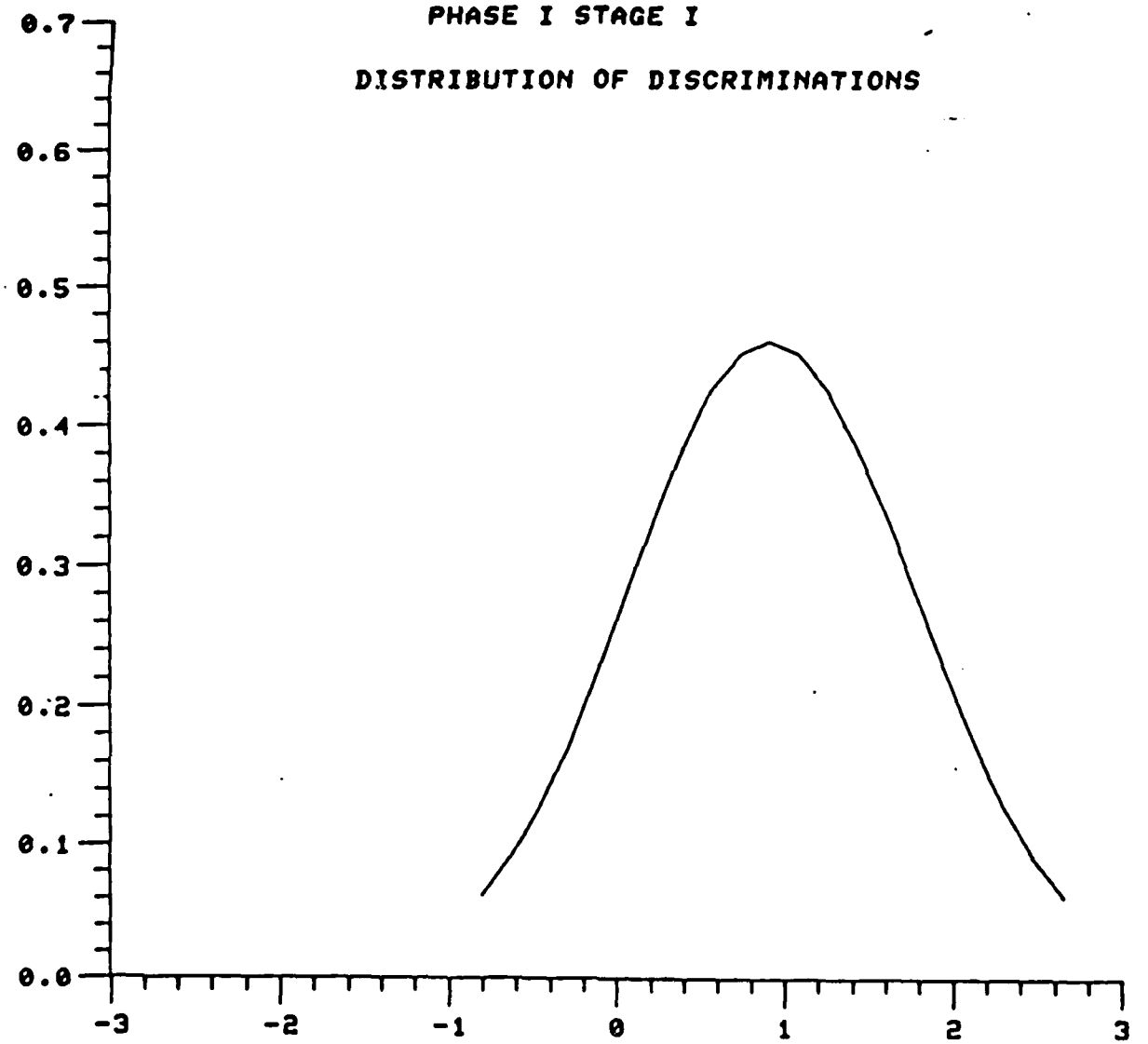
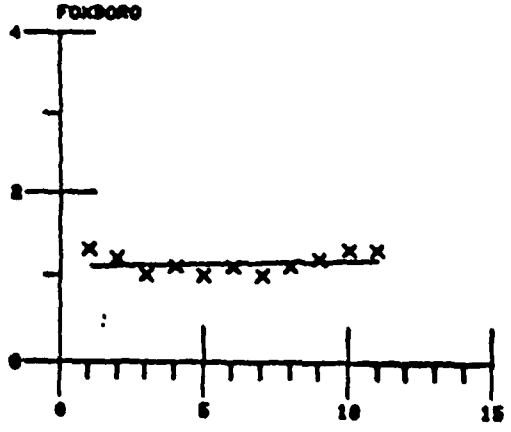
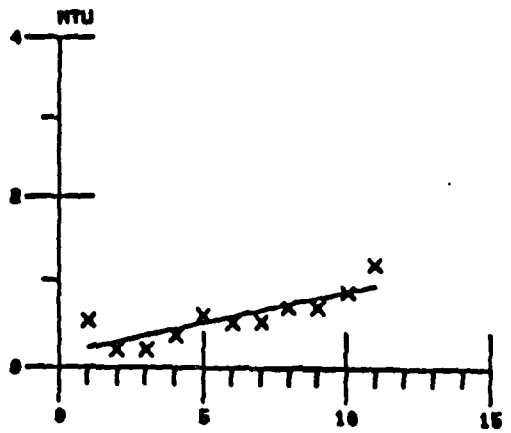

PHASE I STAGE I
DISTRIBUTION OF DISCRIMINATIONS
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-3
-2
-1
0
1
2
3

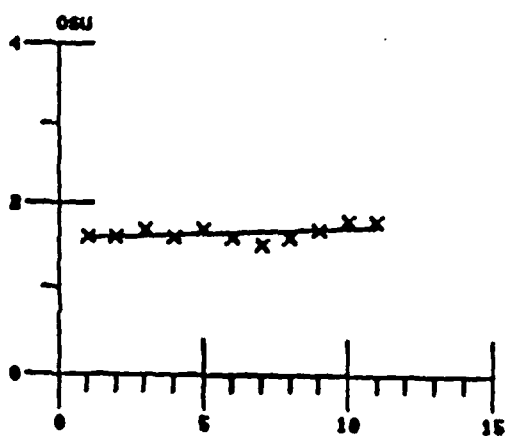
FIGURE 33



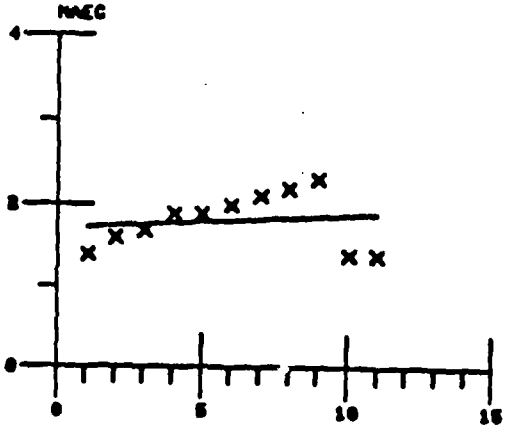
Y = 0.000 X + 1.091
YBAR = 1.145 SY = 0.121
SYX = 0.124 R = 0.000



Y = 0.074 X + 0.139
YBAR = 0.582 SY = 0.290
SYX = 0.164 R = 0.825



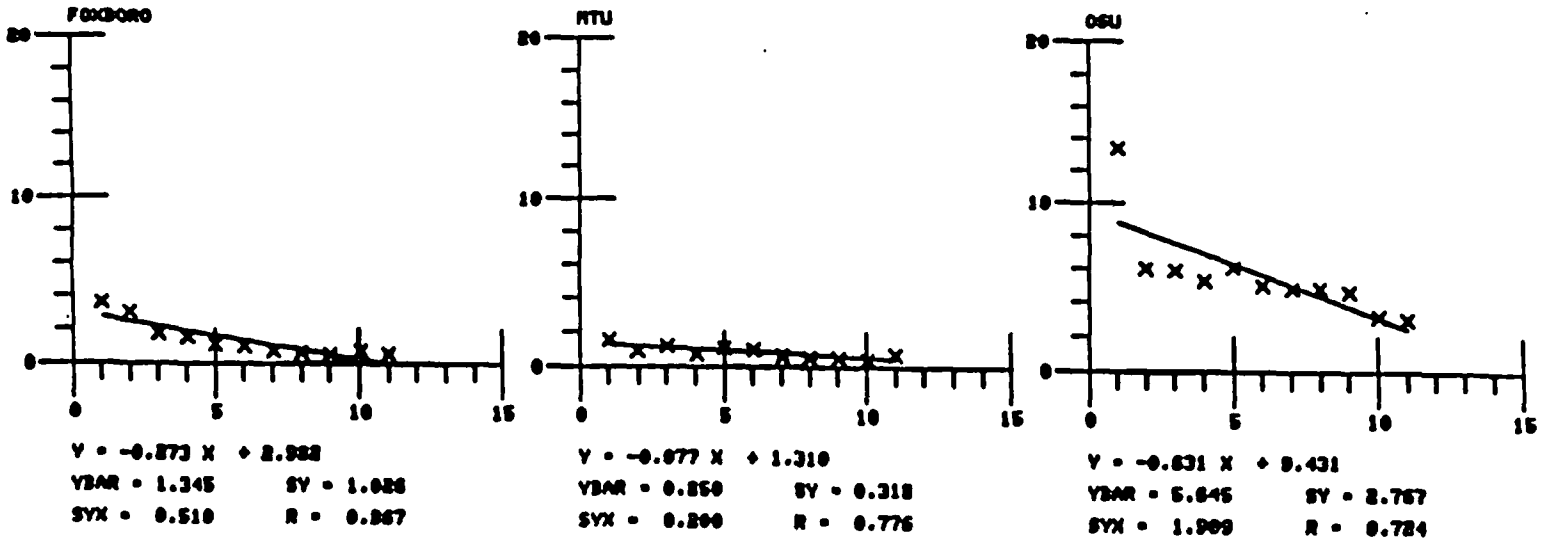
Y = 0.015 X + 1.567
YBAR = 1.655 SY = 0.093
SYX = 0.084 R = 0.430

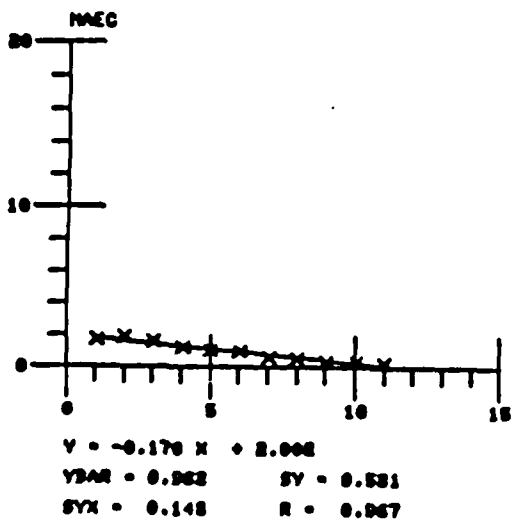


Y = 0.016 X + 1.711
YBAR = 1.809 SY = 0.330
SYX = 0.343 R = 0.000

NTI
PHASE I STAGE II PH887
FERROGRAPHY DATA
DENSITY VS SLIDE POSITION
NTU MINERAL
LIGHT
REFLECTED
CONVENTIONAL

FIGURE 34





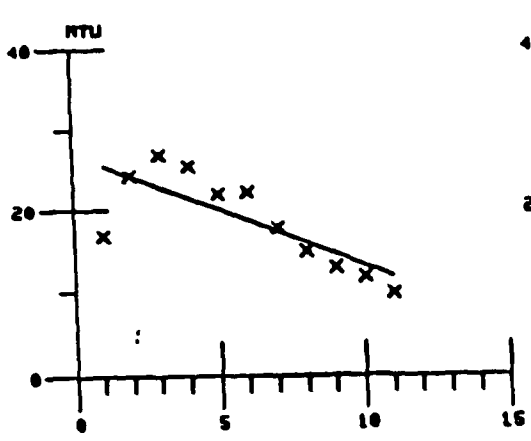
PHASE I STAGE II MTI PH887

FERROGRAPHY DATA

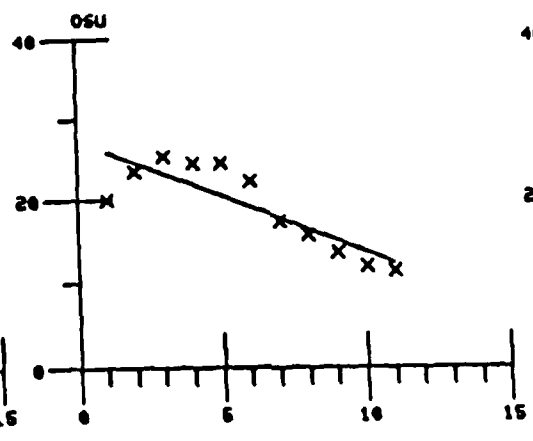
DENSITY VS SLIDE POSITION

NAEC SYNTHETIC
MEDIUM
TRANSMITTED
NEW METHOD

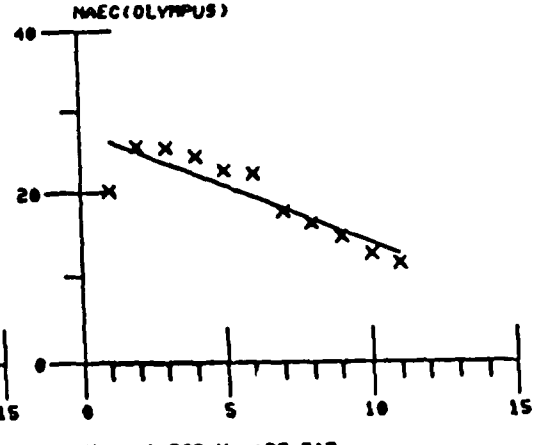
FIGURE 35



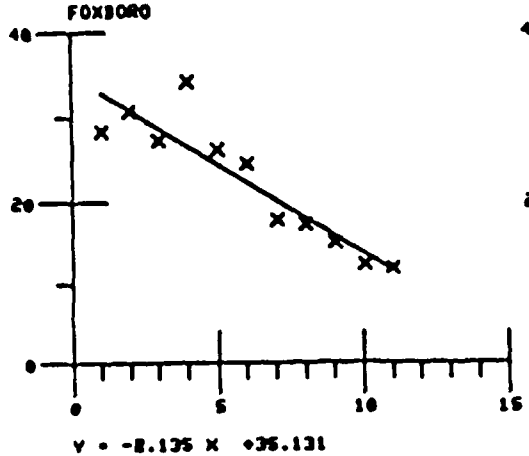
Y = -1.381 X +26.749
YBAR =18.464 SY = 5.852
SYX = 3.839 R = 0.755



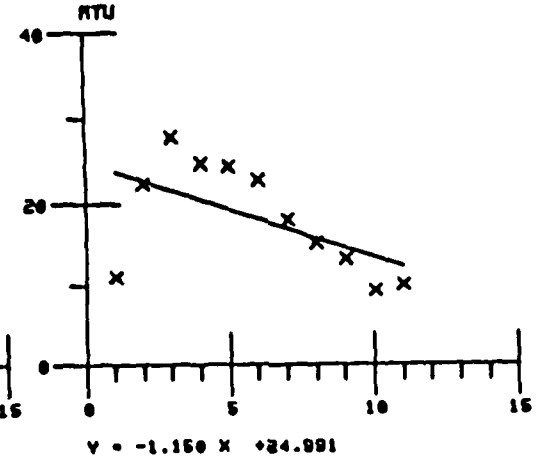
Y = -1.366 X +27.180
YBAR =18.882 SY = 6.331
SYX = 2.960 R = 0.832



Y = -1.365 X +27.515
YBAR =19.327 SY = 5.164
SYX = 2.622 R = 0.862



Y = -8.135 X +35.131
YBAR =22.318 SY = 7.746
SYX = 3.306 R = 0.904



Y = -1.150 X +24.991
YBAR =18.891 SY = 6.665
SYX = 5.761 R = 0.503

PHASE I STAGE II MTI PN887

FERROGRAPHY DATA

DENSITY VS SLIDE POSITION

MTU MINERAL
HEAVY
TRANSMITTED
NEW METHOD

C. Lighting Techniques

1) Mean of reflected higher than transmitted.

D. Indexing Technique

1) Mean and standard deviation very similar.

4.2.2 Coefficient of Variation

A. Interlaboratory as presented in Table 3

1) Range $\simeq$ 16 - 93%

Mean $\simeq$ 57%

2) Sample Type

(a) Slight variation between types of fluid.

3) Sample Concentration

(a) Light and heavy concentrations most significant.

- Heavy Mean COV $\simeq$ 60%
- Medium Mean COV $\simeq$ 22%
- Light Mean COV $\simeq$ 89%

4) Slide Position

(a) COV consistent over slide position.

4.2.3 Graphical Regression Analysis

Representative Phase I - Stage II regression analysis plots are presented in Figures 36, 37, and 38.

The following results can be drawn from the analysis of the total regression analysis plot population.

A. Poor quantitative interlaboratory correlation exists.

B. Inconsistent data bias exists.

C. Poor discrimination as presented in Figure 39

Mean $\simeq 1.52$

D. Substantial scatter exists.

TABLE 3

PHASE I STAGE II - COEFFICIENT OF VARIATION INTERLABORATORY

SLIDE POSITION	MEAN	STD. DEVIATION	COEFFICIENT OF VARIATION
SLIDE I.D.: MTU/MINERAL/MEDIUM 4095			
1	0.4115E+01	0.1233E+01	0.2997E+02
5	0.4680E+01	0.7689E+00	0.1643E+02
10	0.4645E+01	0.1084E+01	0.2334E+02
SLIDE I.D.: NAEC/SYN/HEAVY F3336			
1	0.1591E+02	0.4778E+01	0.3004E+02
5	0.1105E+02	0.7410E+01	0.6705E+02
10	0.7262E+01	0.6191E+01	0.8525E+02
SLIDE I.D.: MTU/MINERAL/LIGHT 4092			
1	0.3082E+01	0.2737E+01	0.8879E+02
5	0.3199E+01	0.2765E+01	0.8643E+02
10	0.4131E+01	0.3855E+01	0.9333E+02

FIGURE 36

PHASE I STAGE II - GRAPHICAL REGRESSION ANALYSIS PLOTS

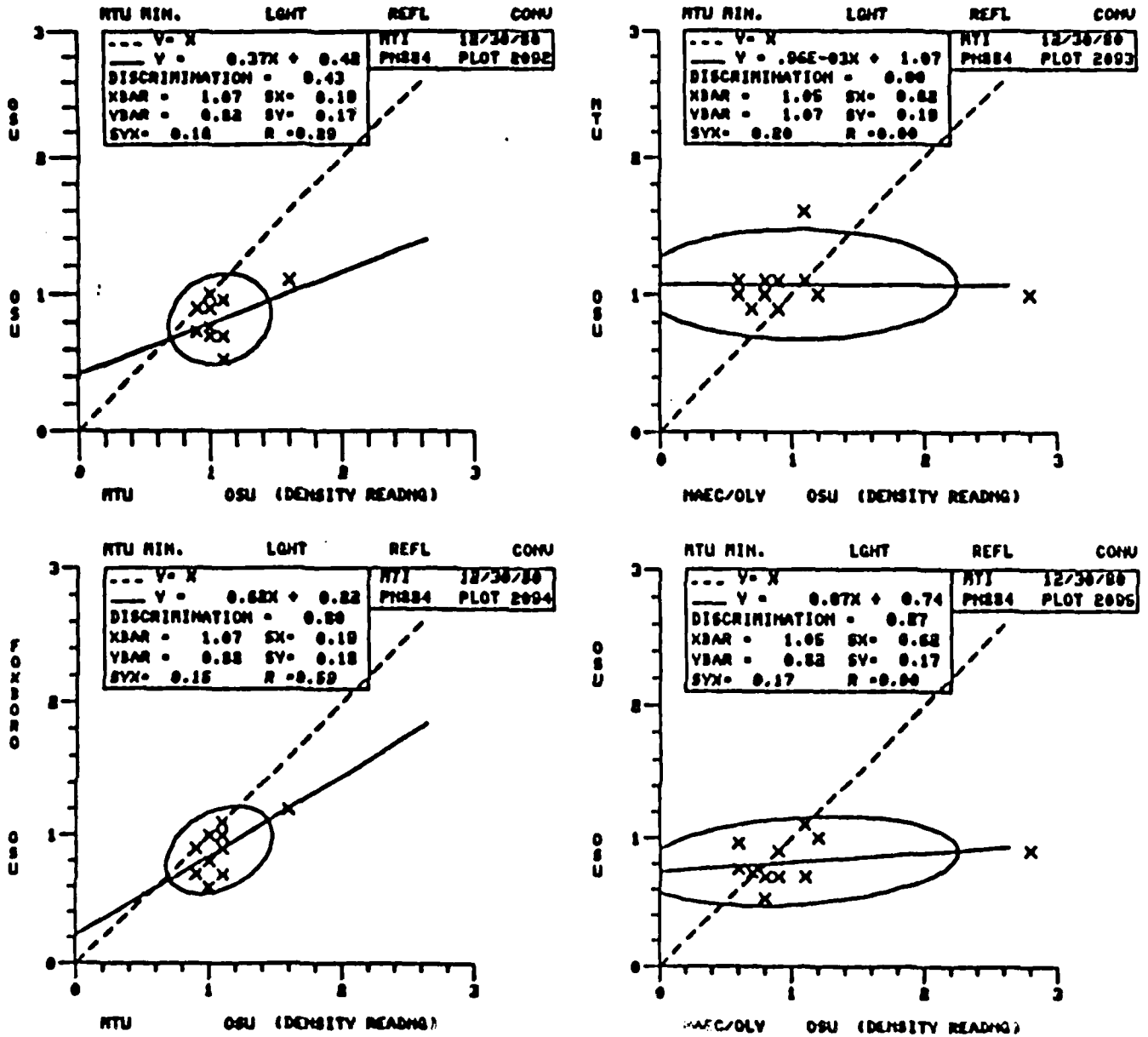


FIGURE 37

PHASE I STAGE II - GRAPHICAL REGRESSION ANALYSIS PLOTS

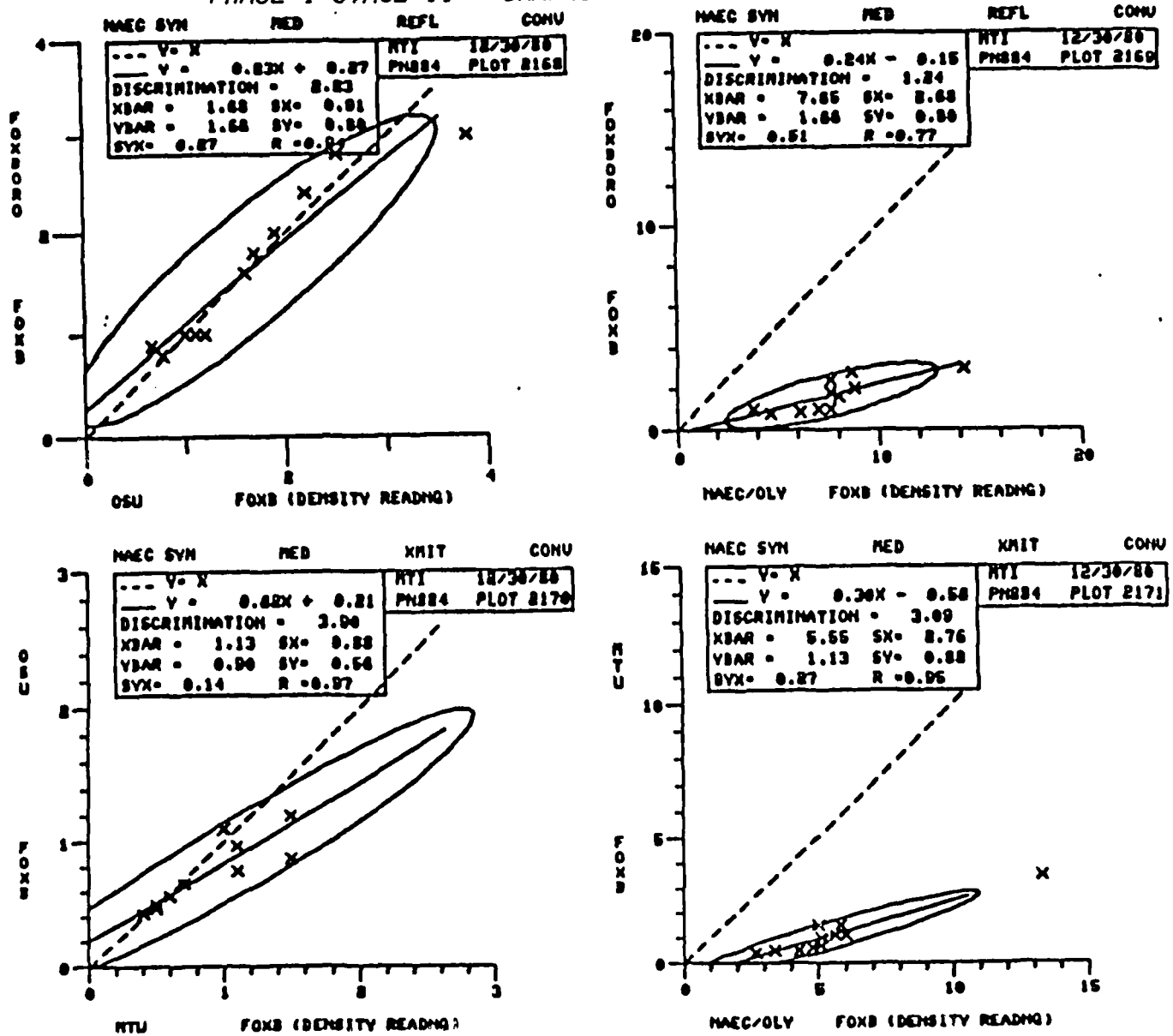


FIGURE 38

PHASE I STAGE II - GRAPHICAL REGRESSION ANALYSIS PLOTS

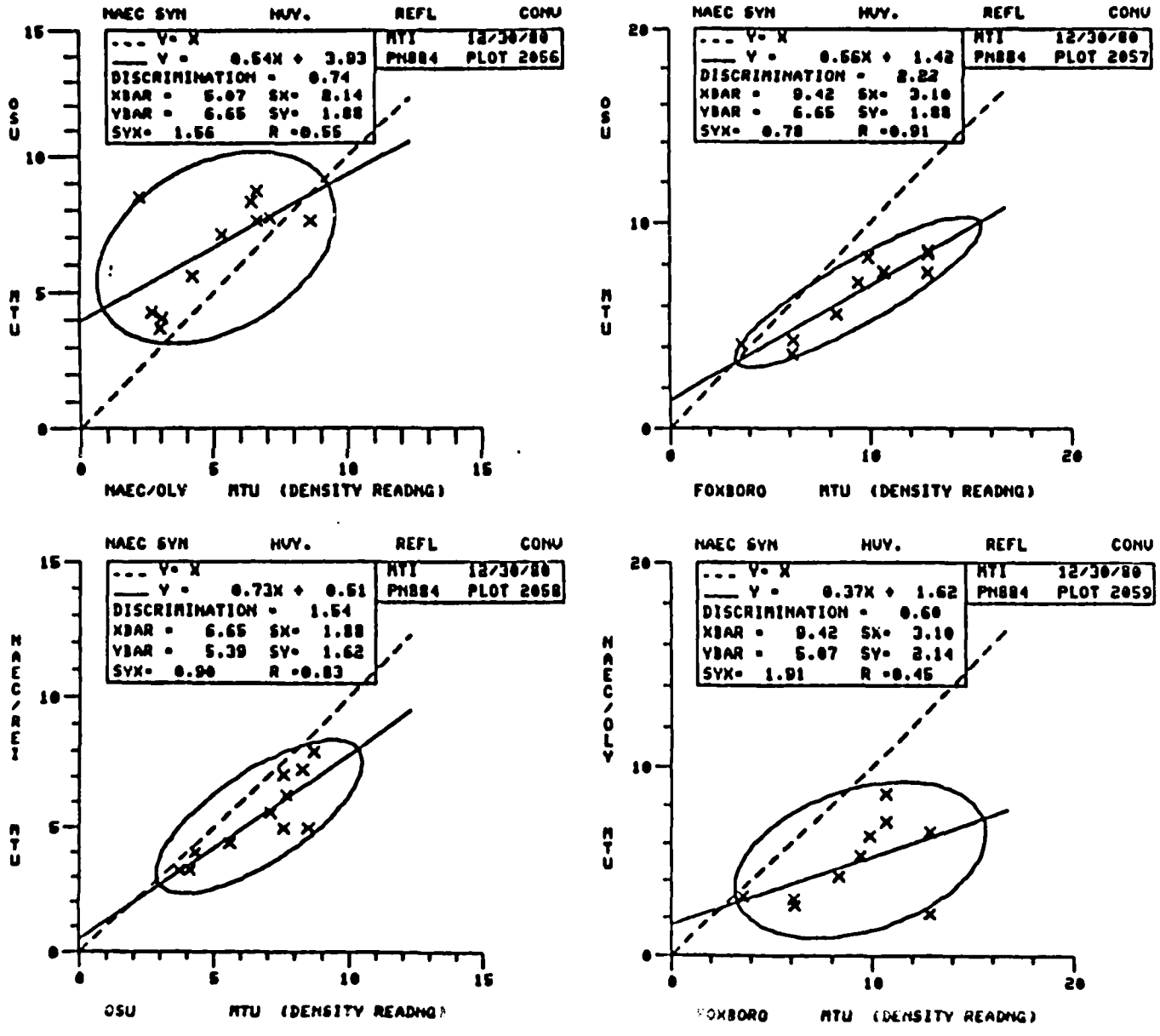
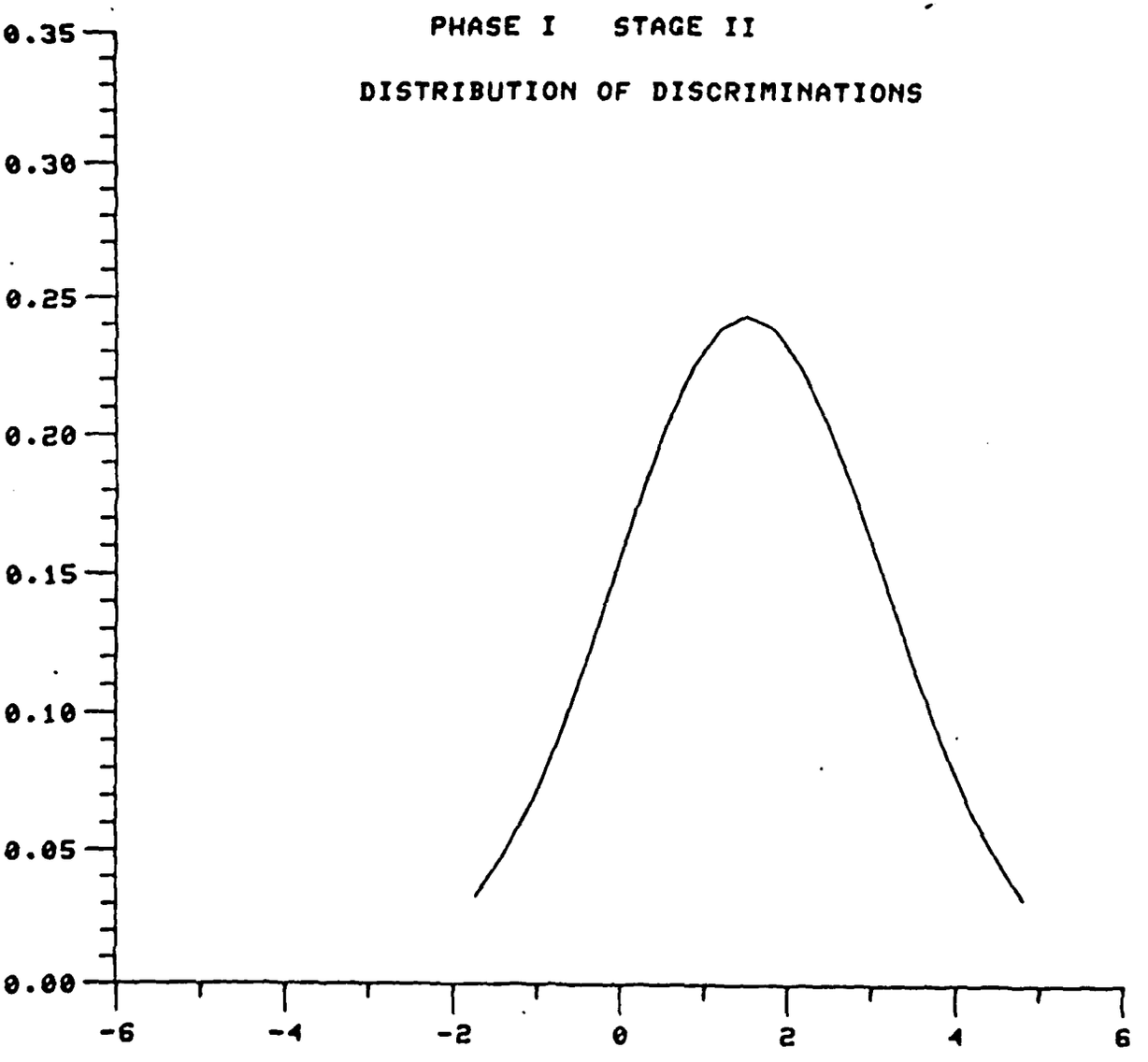


FIGURE 39


PHASE I STAGE II
DISTRIBUTION OF DISCRIMINATIONS
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-6
-4
-2
0
2
4
6

4.3 Phase II - Stage I

Results from the third verification program stage are presented in the following summary. As described previously, this stage involved the analysis of fluid samples.

4.3.1 Data Plots

Representative Phase II - Stage I data plots are presented in Figures 40, 41, 42, and 43.

The following results can be drawn from the analysis of the total plot population.

A. Trending agrees.

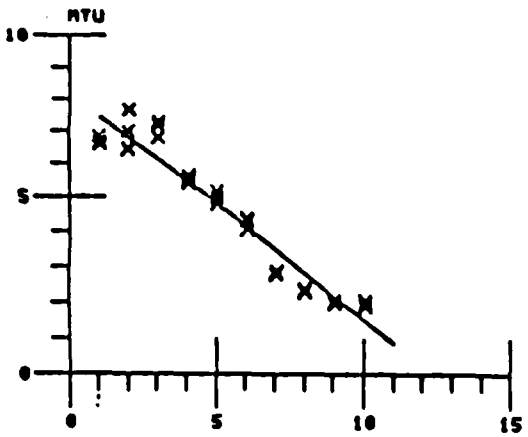
B. Quantitative variations exist.

C. Sample Type as presented in Figure 44
   1) Low Debris Concentration Levels
      Mineral Mean Density $\simeq$ 3.8
      Hydraulic Mean Density $\simeq$ 7.8
      Synthetic Mean Density $\simeq$ 4.3

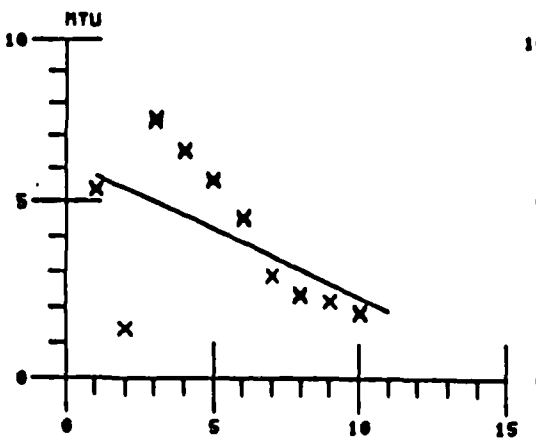
D. Laboratories as presented in Figure 45
   1) Mean density value and standard deviation of NAEC, MTU, and OSU similar
      Mean Density $\simeq$ 4.4
      Standard Deviation $\simeq$ 2.4
   2) Mean density value and standard deviation of Foxboro and JOAP/TSC similar
      Mean Density $\simeq$ 7.0
      Standard Deviation $\simeq$ 5.3

E. Slide Position as presented in Figures 46, 47, and 48
   1) Observations consistent over slide position

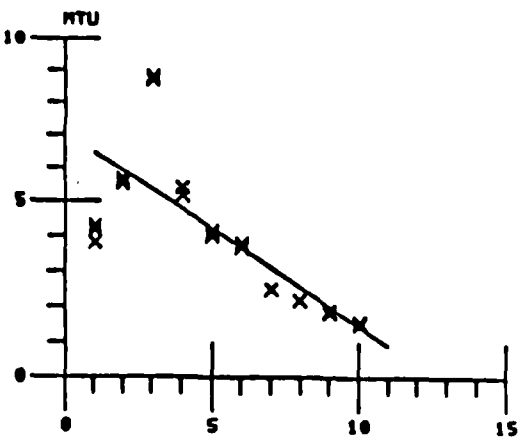
FIGURE 40



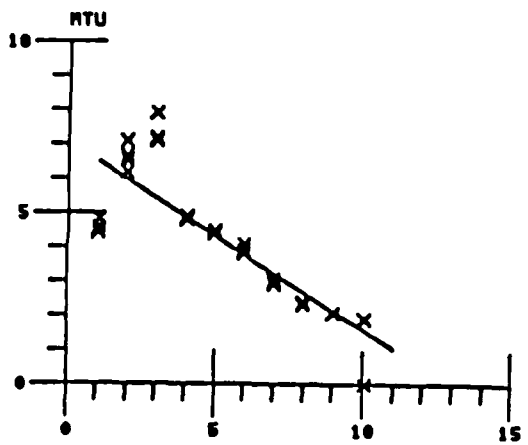
Y = -0.659 X + 8.096
YBAR = 4.474 SY = 1.986
SYX = 0.547 R = 0.961



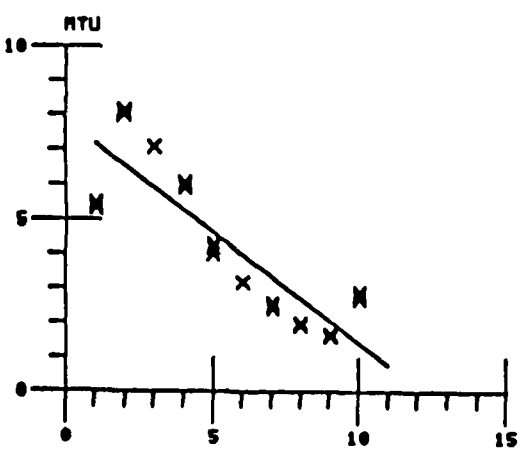
Y = -0.388 X + 6.167
YBAR = 4.034 SY = 2.076
SYX = 1.763 R = 0.528



Y = -0.553 X + 7.003
YBAR = 3.962 SY = 2.093
SYX = 1.358 R = 0.761



Y = -0.545 X + 7.059
YBAR = 4.060 SY = 1.848
SYX = 0.949 R = 0.857



Y = -0.642 X + 7.823
YBAR = 4.290 SY = 2.163
SYX = 1.109 R = 0.859

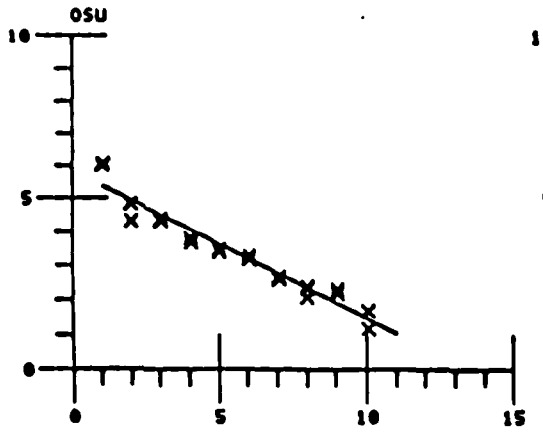
PHASE II STAGE I MTI PH887

FERROGRAPHY DATA

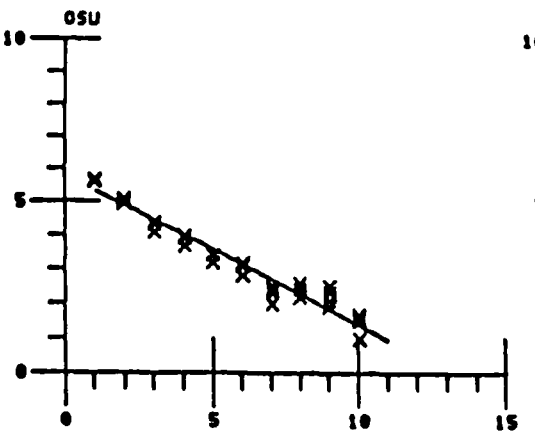
DENSITY US SLIDE POSITION

MAEC SYNTHETIC

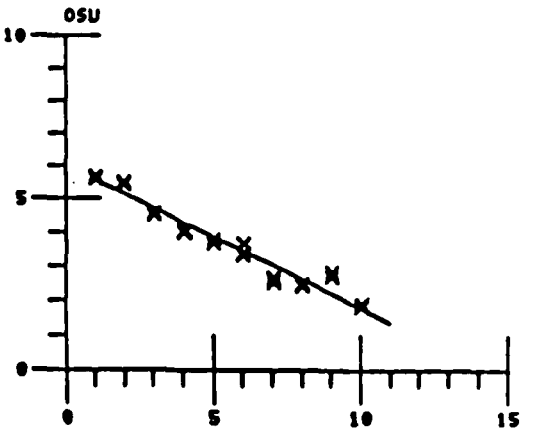
FIGURE 41



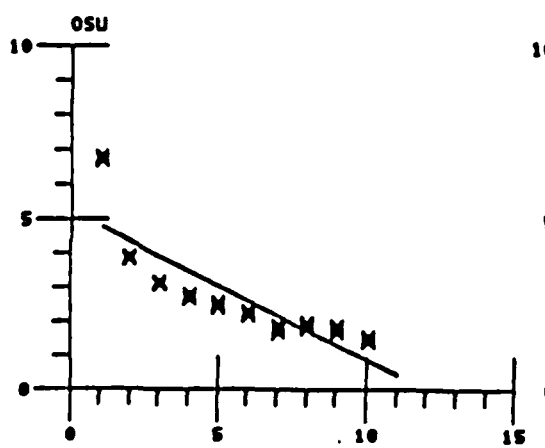
Y = -0.429 X + 5.781
YBAR = 3.424 SY = 1.281
SYX = 0.389 R = 0.971



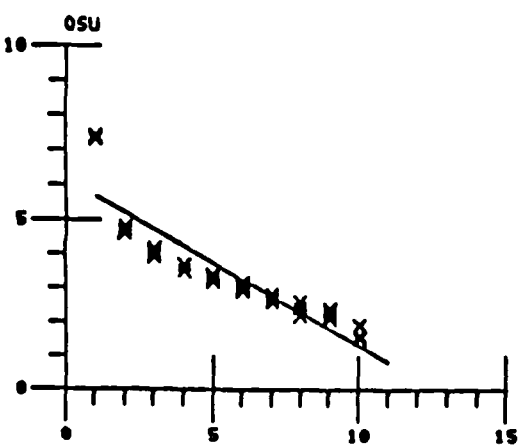
Y = -0.437 X + 5.768
YBAR = 3.364 SY = 1.298
SYX = 0.280 R = 0.976



Y = -0.419 X + 5.960
YBAR = 3.658 SY = 1.246
SYX = 0.282 R = 0.974



Y = -0.432 X + 5.209
YBAR = 2.836 SY = 1.495
SYX = 0.825 R = 0.834



Y = -0.487 X + 6.167
YBAR = 3.488 SY = 1.570
SYX = 0.692 R = 0.898

MTI

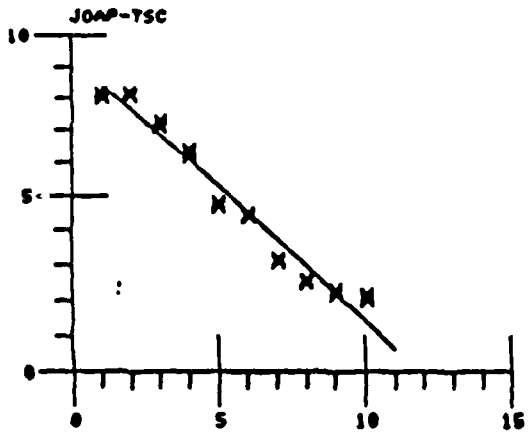
PHASE II STAGE I PN887

FERROGRAPHY DATA

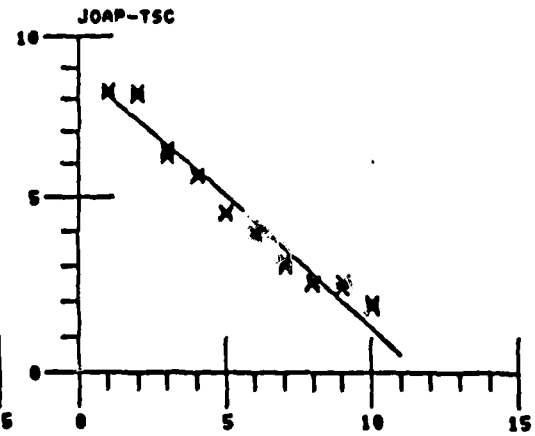
DENSITY VS SLIDE POSITION

NAEC SYNTHETIC

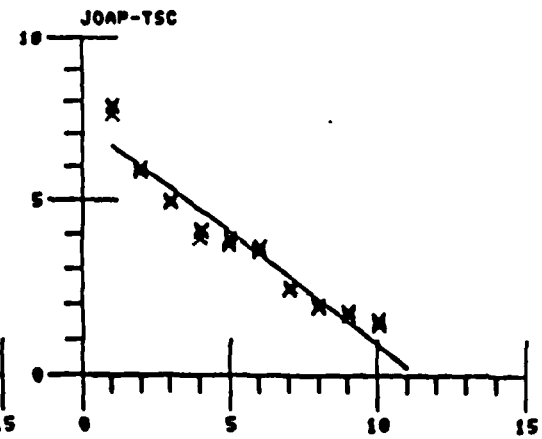
FIGURE 42



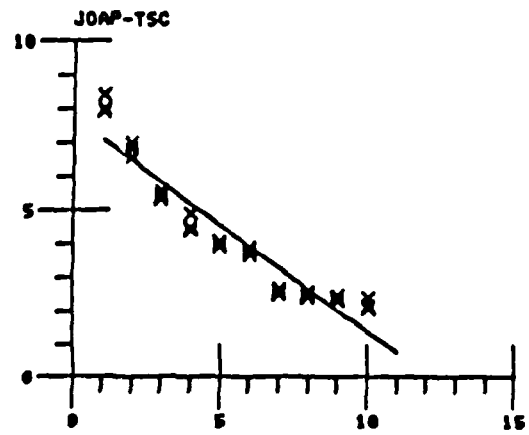
Y = -0.779 X + 9.191
YBAR = 4.988 SY = 2.302
SYX = 0.444 R = 0.981



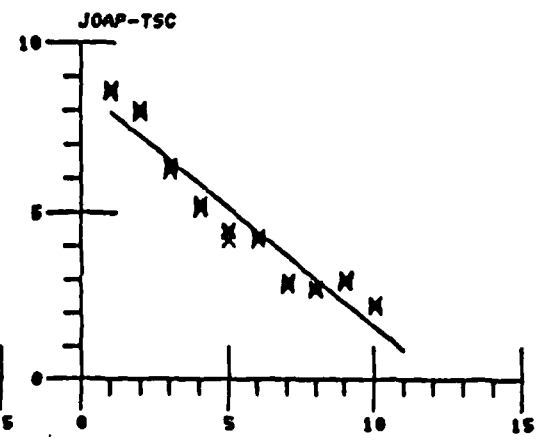
Y = -0.760 X + 8.869
YBAR = 4.692 SY = 2.254
SYX = 0.476 R = 0.977



Y = -0.641 X + 7.296
YBAR = 3.778 SY = 1.933
SYX = 0.530 R = 0.962



Y = -0.630 X + 7.707
YBAR = 4.242 SY = 1.920
SYX = 0.695 R = 0.951



Y = -0.706 X + 8.653
YBAR = 4.770 SY = 2.139
SYX = 0.623 R = 0.957

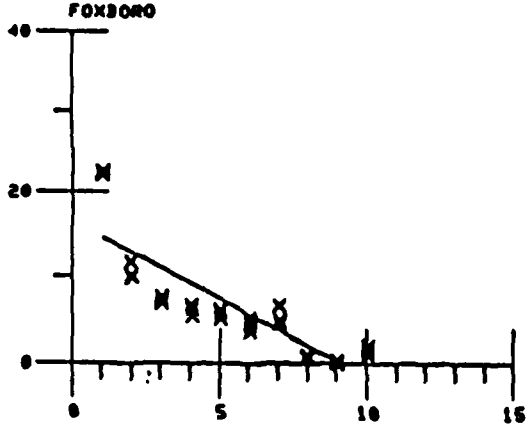
PHASE II STAGE I RTI PN887

FERROGRAPHY DATA

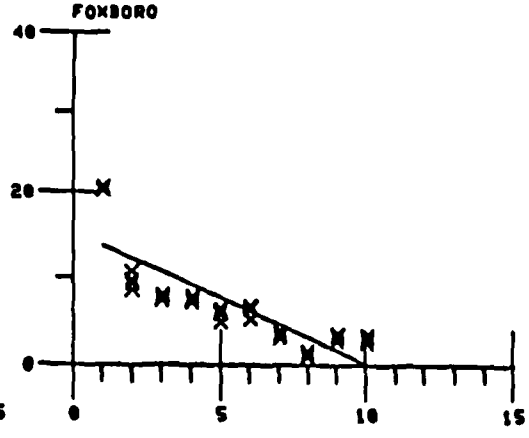
DENSITY US SLIDE POSITION

MAEC SYNTHETIC

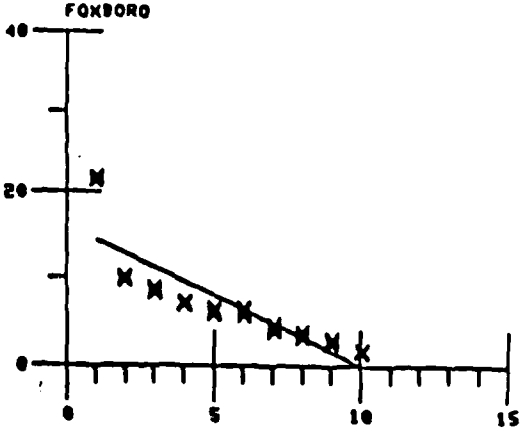
FIGURE 43



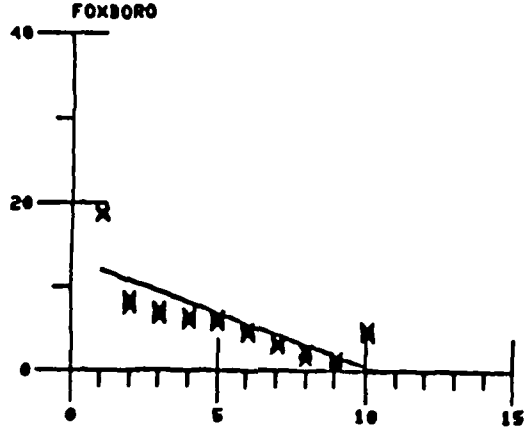
Y = -1.798 X +16.328
YBAR = 6.442 SY = 6.193
SYX = 3.374 R = 0.839



Y = -1.491 X +15.055
YBAR = 6.852 SY = 5.229
SYX = 2.965 R = 0.824



Y = -1.607 X +15.988
YBAR = 7.158 SY = 5.494
SYX = 2.937 R = 0.845



Y = -1.271 X +13.187
YBAR = 6.194 SY = 4.723
SYX = 2.988 R = 0.776

PHASE II STAGE I MTI PH887

FERROGRAPHY DATA

DENSITY VS SLIDE POSITION

NAEC SYNTHETIC

FIGURE 44

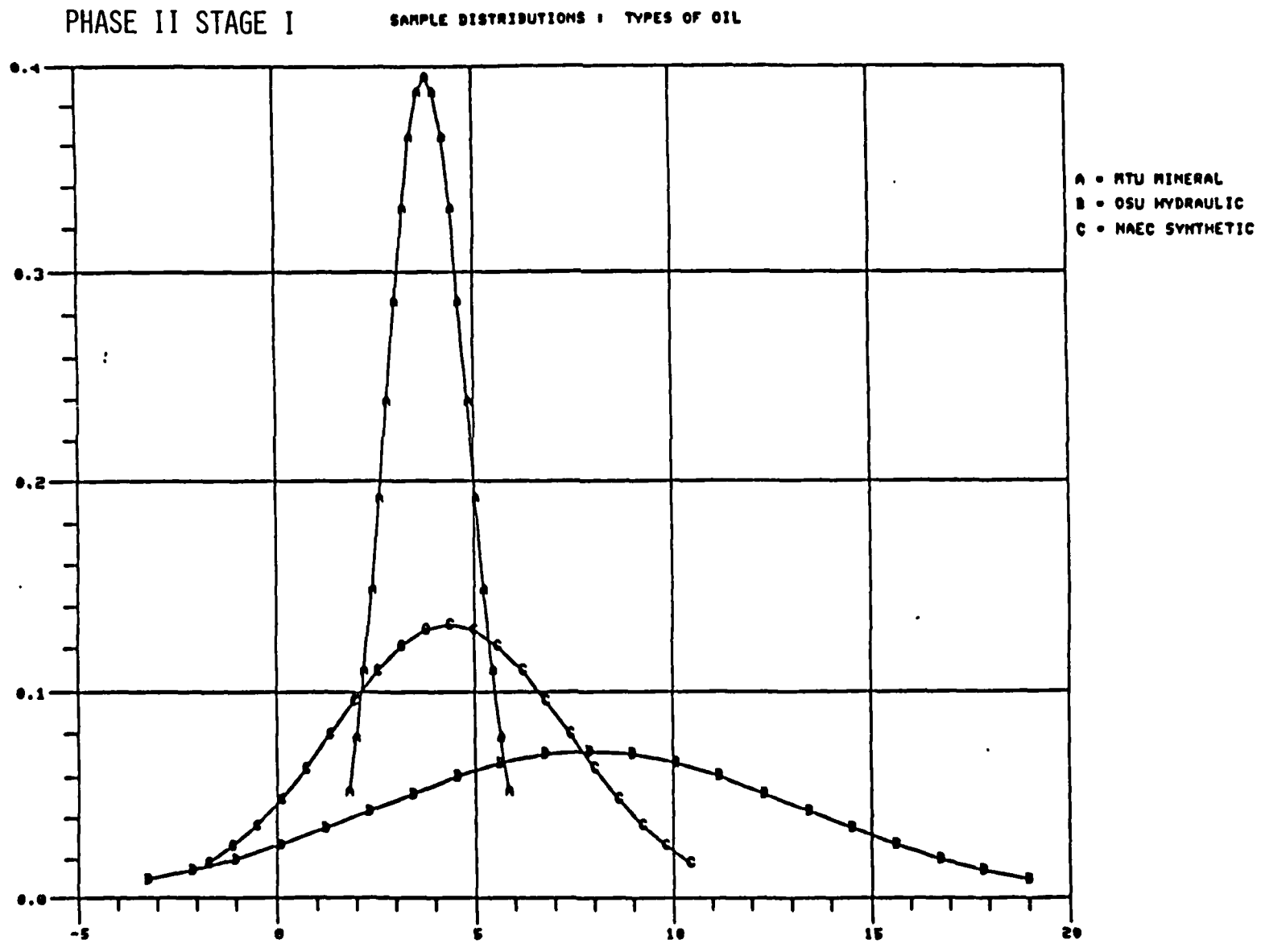

PHASE II STAGE I
SAMPLE DISTRIBUTIONS : TYPES OF OIL
A • MTU MINERAL
B • OSU HYDRAULIC
C • NAEC SYNTHETIC
0.4
0.3
0.2
0.1
0.0
-5
0
5
10
15
20

FIGURE 45

PHASE II STAGE I SAMPLE DISTRIBUTIONS: LABORATORIES

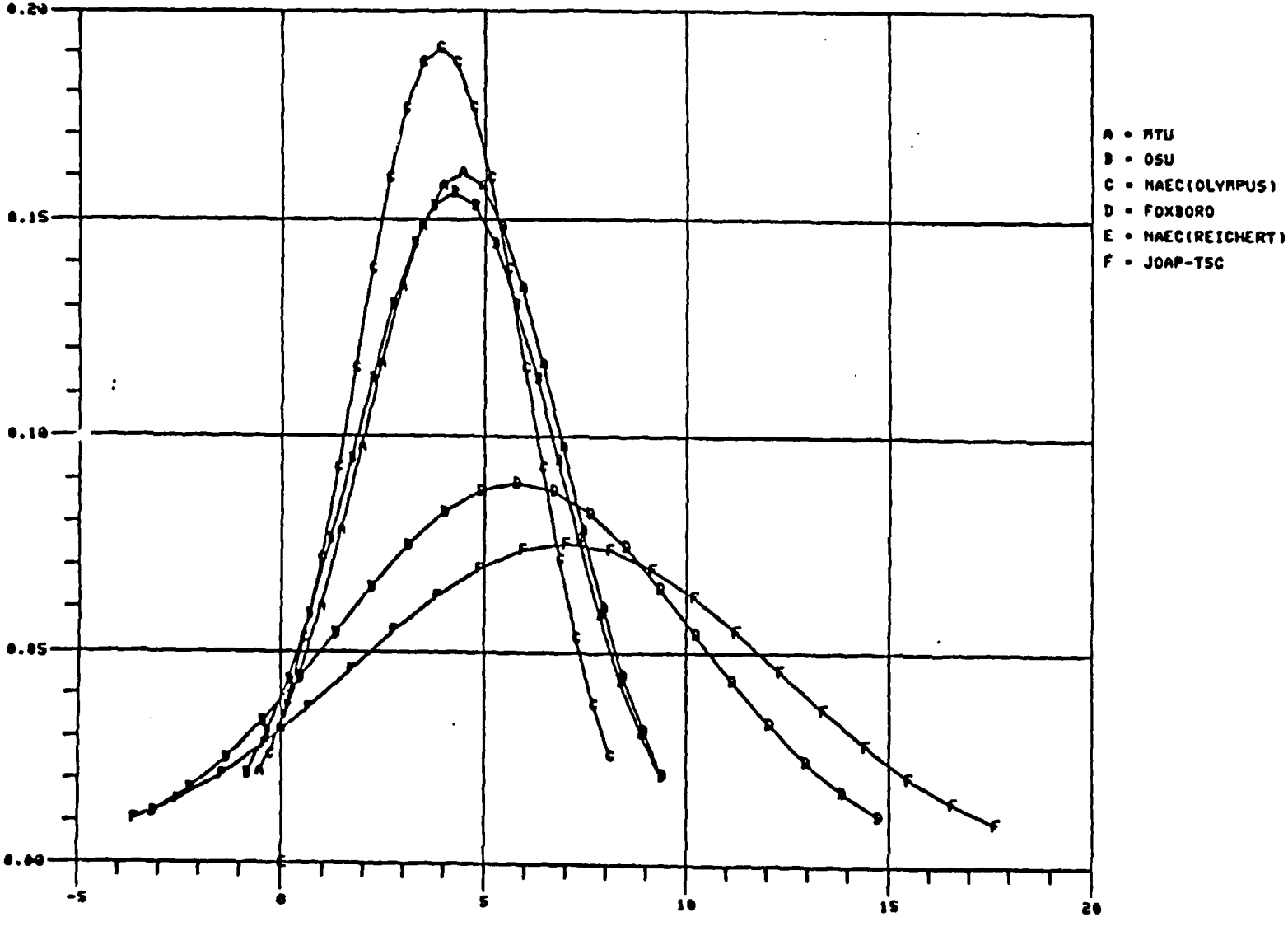


FIGURE 46

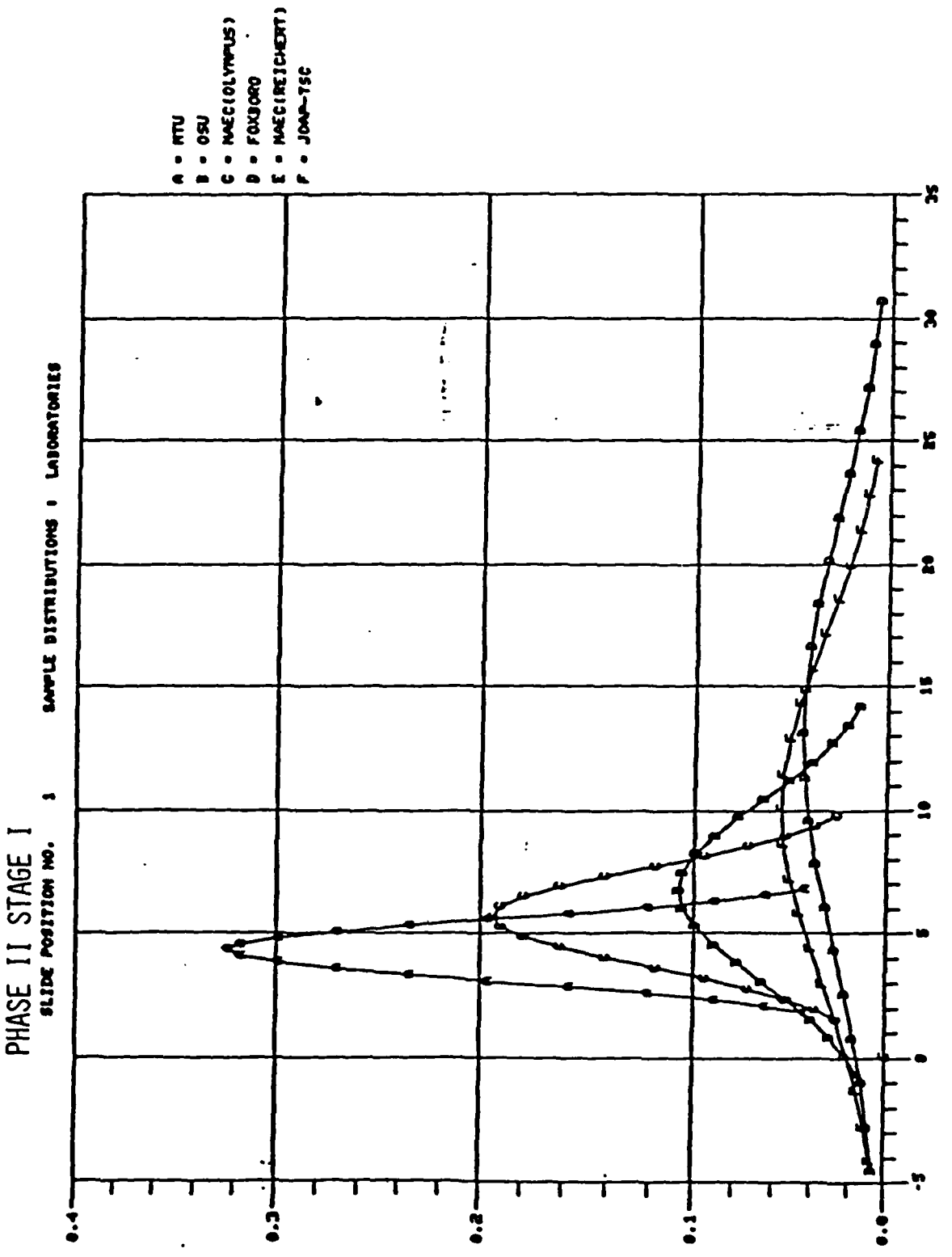

PHASE II STAGE I
SLIDE POSITION NO. 1 SAMPLE DISTRIBUTIONS : LABORATORIES
A = RTU
B = OSU
C = NAEC(OLYMPUS)
D = FOXBORO
E = NAEC(REICHERT)
F = JOAN-TSC
0.4
0.3
0.2
0.1
0.0
-5
0
5
10
15
20
25
30

FIGURE 47

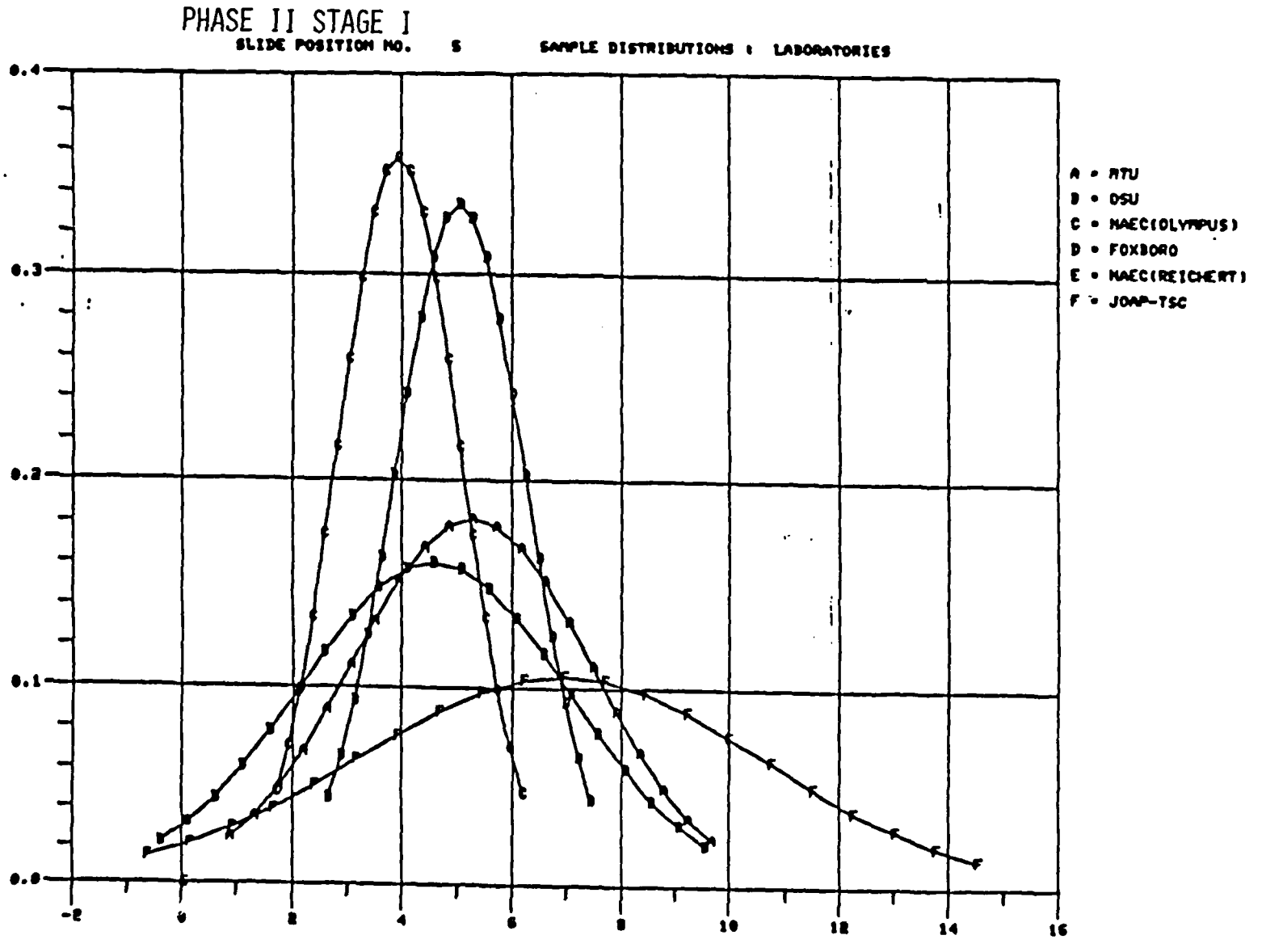
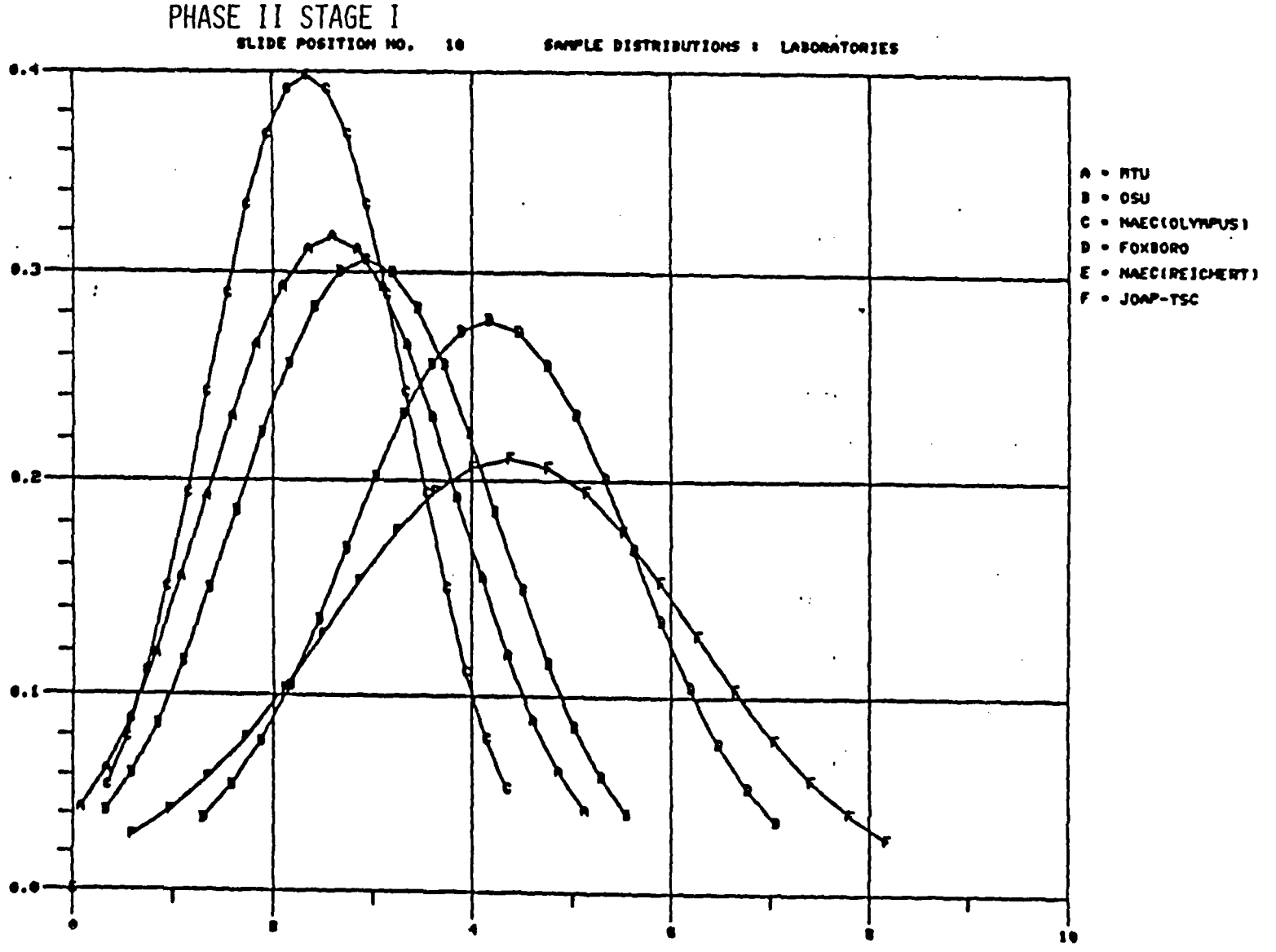

PHASE II STAGE I
SLIDE POSITION NO. 5
SAMPLE DISTRIBUTIONS : LABORATORIES
0.4
0.3
0.2
0.1
0.0
-2
0
2
4
6
8
10
12
14
16
A = RTU
B = OSU
C = MAEC(OLYMPUS)
D = FOXBORO
E = MAEC(REICHERT)
F = JOAP-TSC

FIGURE 48


PHASE II STAGE I
SLIDE POSITION NO. 10
SAMPLE DISTRIBUTIONS : LABORATORIES
0.4
0.3
0.2
0.1
0.0
0
2
4
6
8
10
A = RTU
B = OSU
C = NAEC(OLYMPUS)
D = FOXBORO
E = NAEC(REICHERT)
F = JOAP-TSC

4.3.2 Coefficient of Variation

A. Intra-Laboratory

1) Range ≃ 0 - 12%

Mean ≃ 5%

2) Sample Type as presented in Figure 49

(a) Mineral COV low with respect to synthetic and hydraulic type samples.

Mineral Mean COV ≃ 3.64

Synthetic Mean COV ≃ 9.01

Hydraulic Mean COV ≃ 13.55

3) Slide Position

(a) COV consistent over slide positions.

4) Laboratory as presented in Figure 50

(a) NAEC, Foxboro, and MTU similar

- Mean COV ≃ 5%

(b) OSU and JOAP/TSC similar

- Mean COV ≃ 2.5%

B. Interlaboratory as presented in Table 4

1) Range ≃ 22-60%

Mean ≃ 37%

2) Sample Type

(a) Mineral COV low with respect to synthetic and hydraulic type samples.

Mineral Mean COV ≃ 26%

Hydraulic Mean COV ≃ 46%

Synthetic Mean COV ≃ 40%

3) Slide Position

(a) COV consistent over slide position

4.3.3 Graphical Regression Analysis

Representative Phase II - Stage I regression analysis plots are presented in Figures 51, 52, and 53.

FIGURE 49

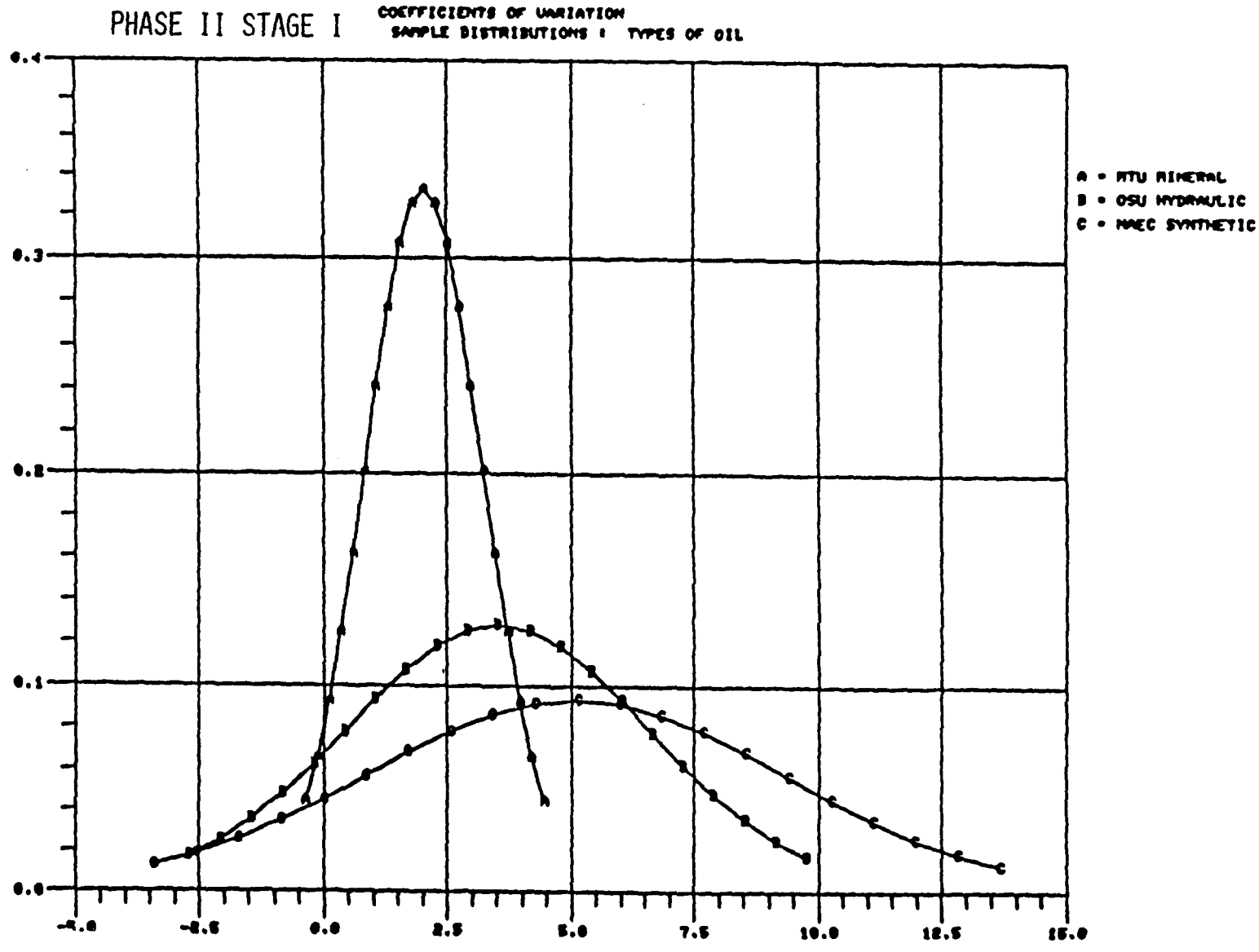

PHASE II STAGE I
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS : TYPES OF OIL
A = MTU MINERAL
B = OSU HYDRAULIC
C = MAEC SYNTHETIC
0.4
0.3
0.2
0.1
0.0
-5.0
-2.5
0.0
2.5
5.0
7.5
10.0
12.5
15.0

FIGURE 50

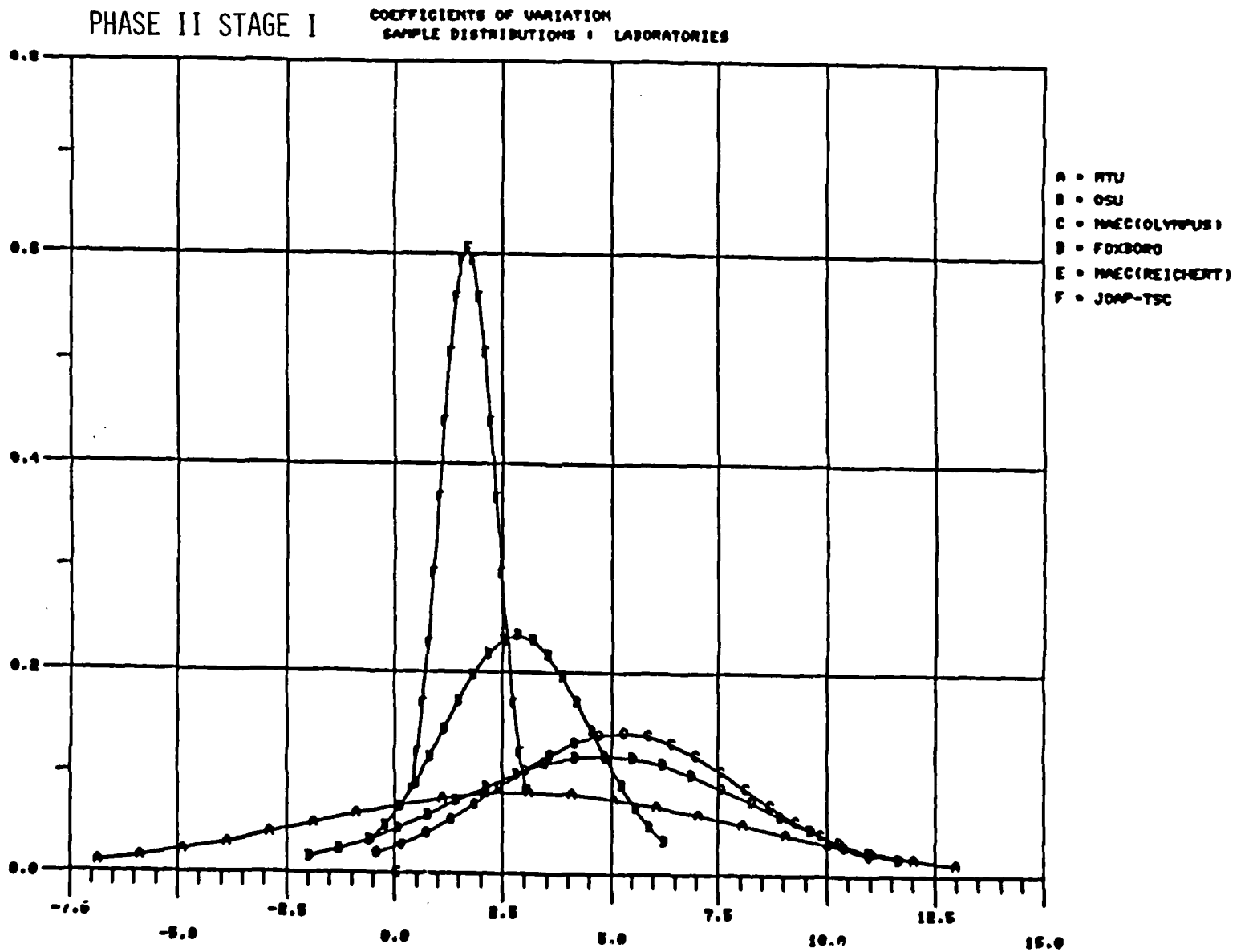

PHASE II STAGE I
COEFFICIENTS OF VARIATION
SAMPLE DISTRIBUTIONS : LABORATORIES
0.8
0.6
0.4
0.2
0.0
-10.0
-5.0
-2.5
0.0
2.5
5.0
7.5
10.0
12.5
15.0
A = RTU
B = OSU
C = MAEC(OLYMPUS)
D = FOXBORO
E = MAEC(REICHERT)
F = JOAP-TSC

TABLE 4

PHASE II STAGE I - COEFFICIENT OF VARIATION - INTERLABORATORY

SLIDE POSITION	TYPE OF OIL	COEFF. OF VARIATION
1	MTU MINERAL	0.3327E+02
5	MTU MINERAL	0.2201E+02
10	MTU MINERAL	0.2401E+02
1	OSU HYDRAULIC	0.5294E+02
5	OSU HYDRAULIC	0.3386E+02
10	OSU HYDRAULIC	0.5572E+02
1	NAEC SYNTHETIC	0.6049E+02
5	NAEC SYNTHETIC	0.2451E+02
10	NAEC SYNTHETIC	0.3847E+02

FIGURE 51

PHASE II STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOT

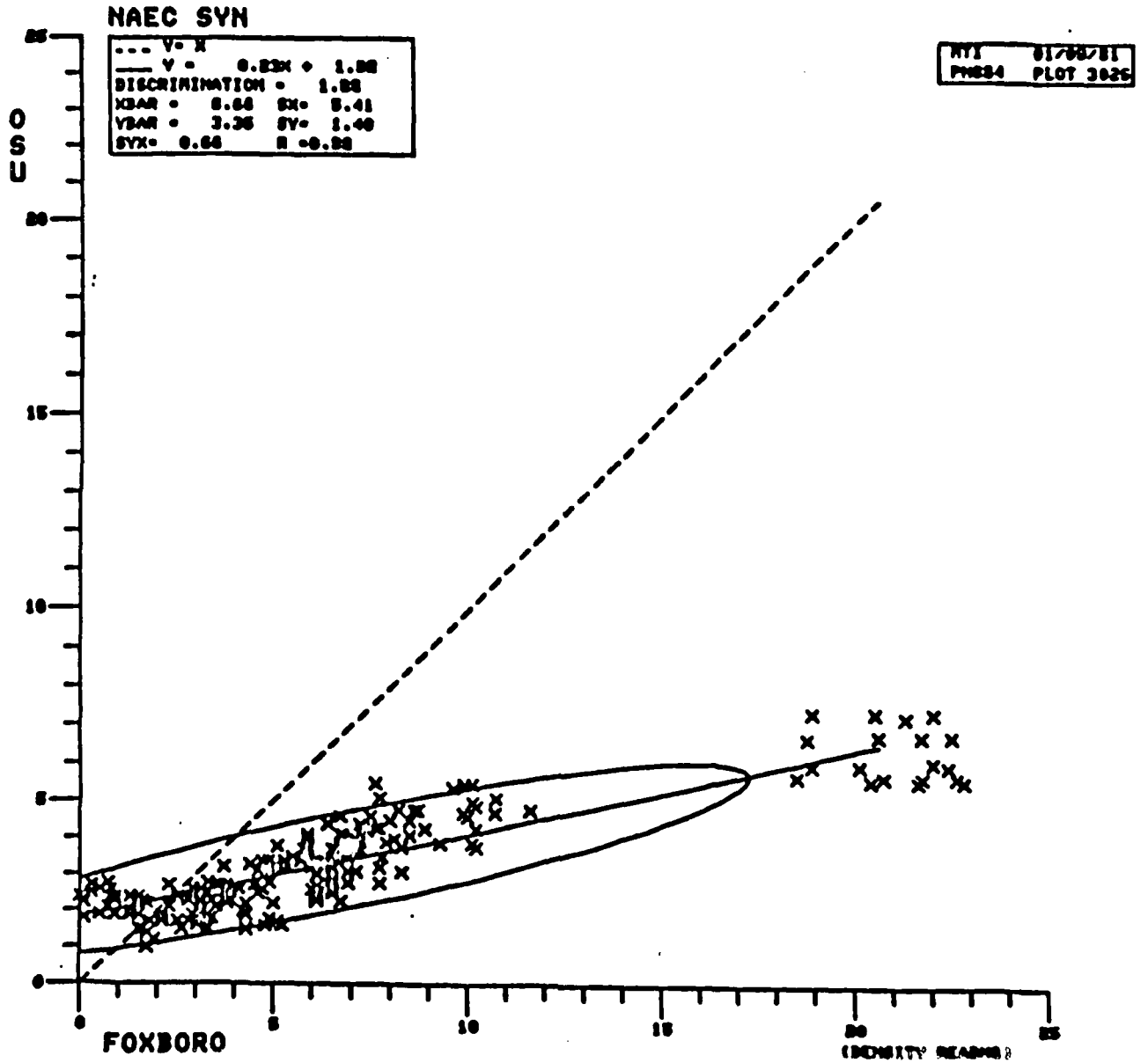


FIGURE 52

PHASE II STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOT

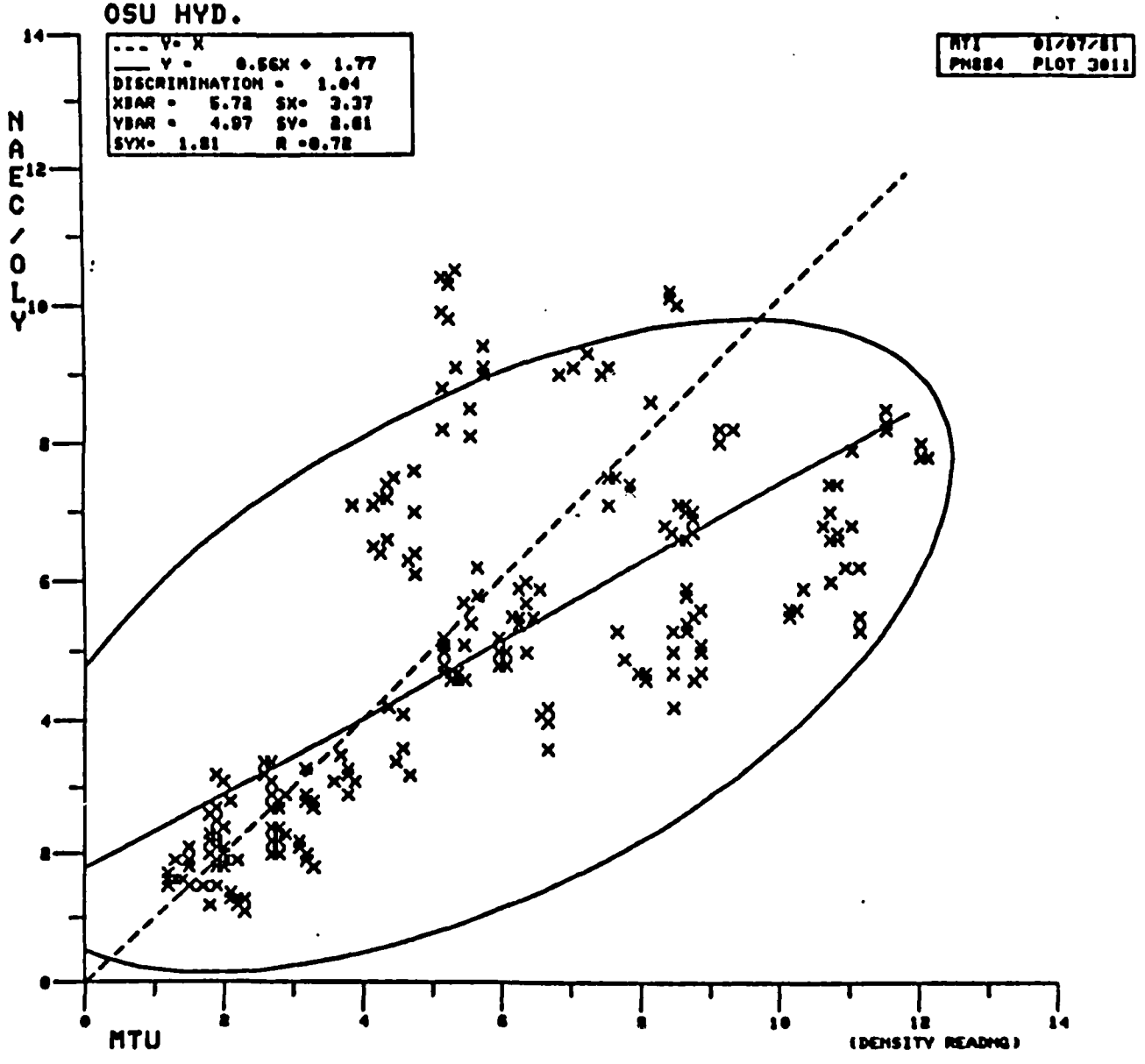
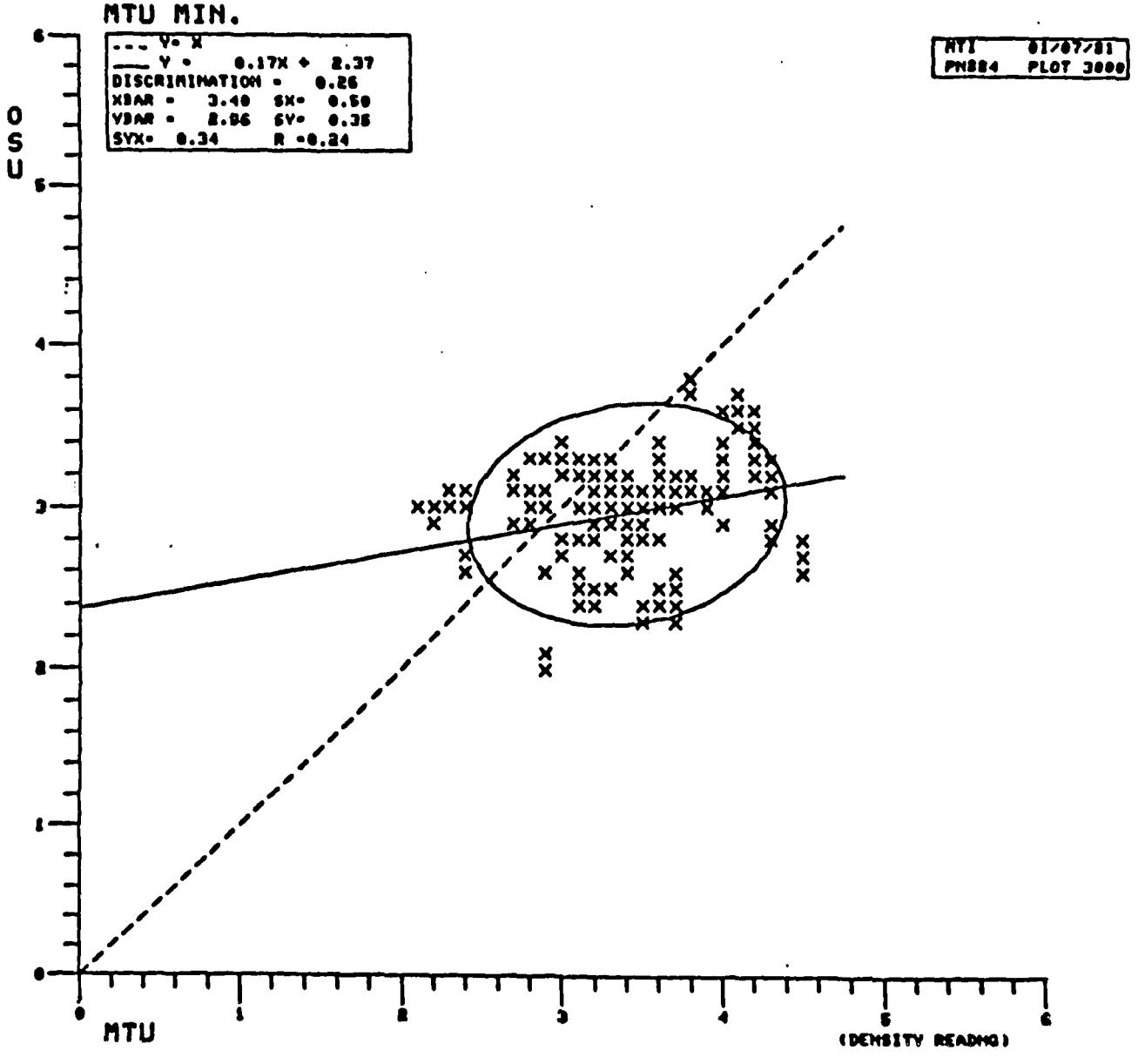


FIGURE 53

PHASE II STAGE I - GRAPHICAL REGRESSION ANALYSIS PLOT



The following results can be drawn from the analysis of the total regression analysis population.

A. Poor quantitative and interlaboratory correlation exists.

B. Inconsistent data bias exists.

C. Poor discrimination as presented in Figure 54.

Mean $\simeq$ .84

D. Substantial scatter exists.

4.4 Phase II - Stage II

Results from the final verification program stage are presented in the following summary. As described previously, this stage involved the analysis of pre-made Ferrogram slides.

4.4.1 Data Plots

Representative Phase II - Stage II data plots are presented in Figures 55, 56, and 57.

The following results can be drawn from the analysis of the total plot population.

A. Trending agrees.

B. Quantitative variations exist.

C. Laboratories as presented in Figure 58.

1) Mean density value very similar for all laboratories

2) Standard deviation very similar for all laboratories.

4.4.2 Coefficient of Variation

A. Intra-Laboratory as presented in Figure 59

1) Range $\simeq$ 0 - 12%

Mean $\simeq$ 4%

FIGURE 54

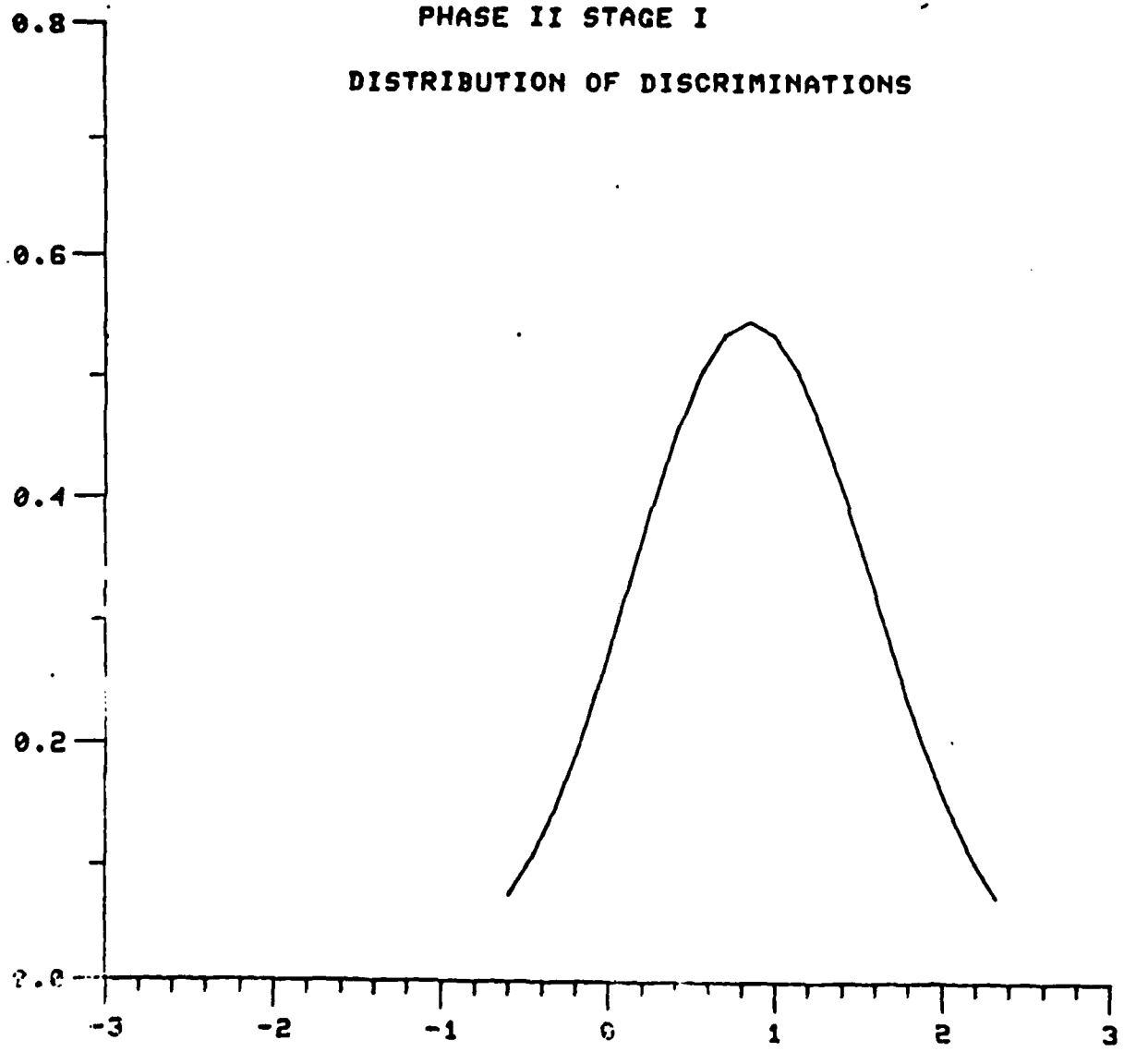

PHASE II STAGE I
DISTRIBUTION OF DISCRIMINATIONS
0.8
0.6
0.4
0.2
0.0
-3
-2
-1
0
1
2
3

FIGURE 55

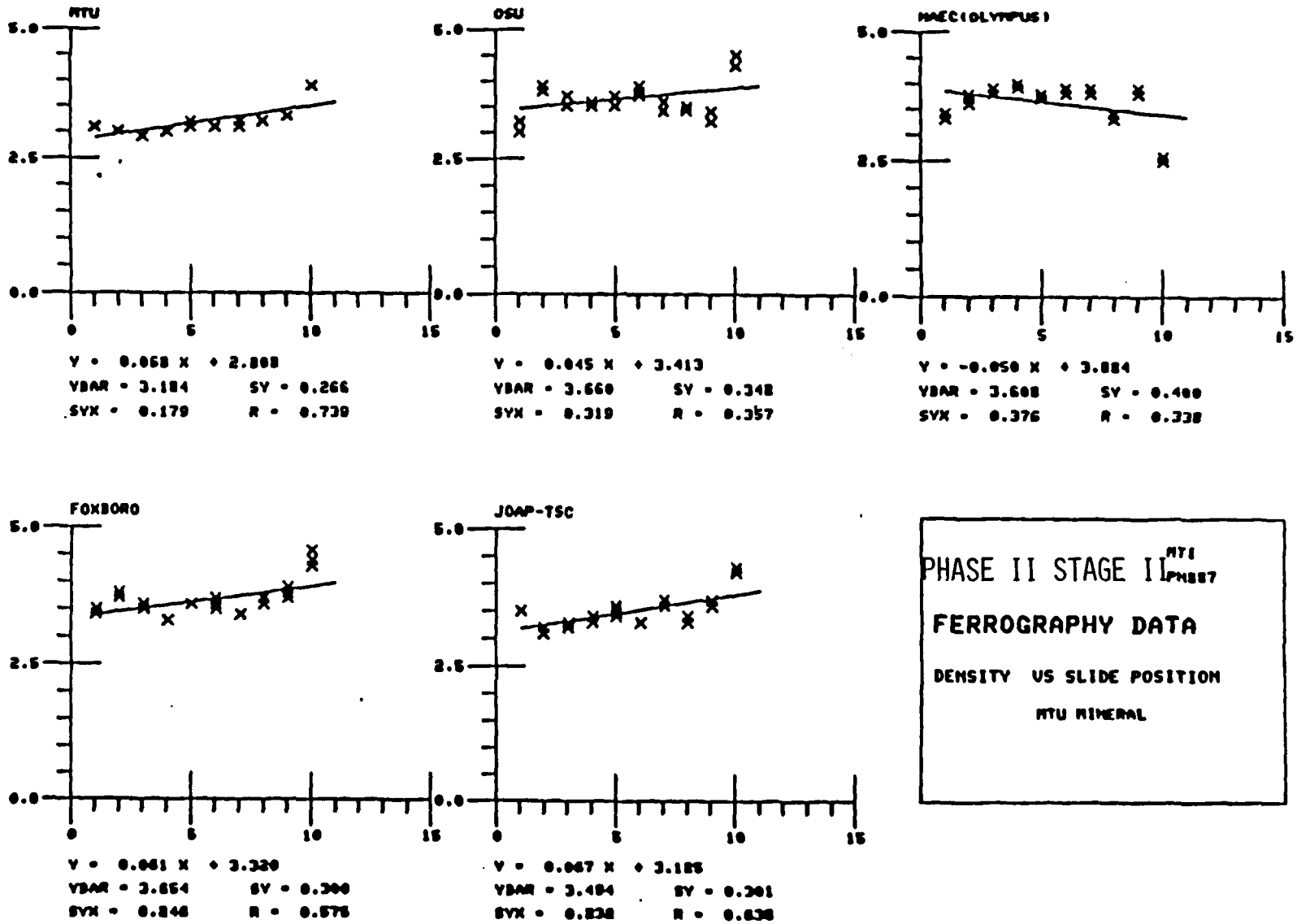
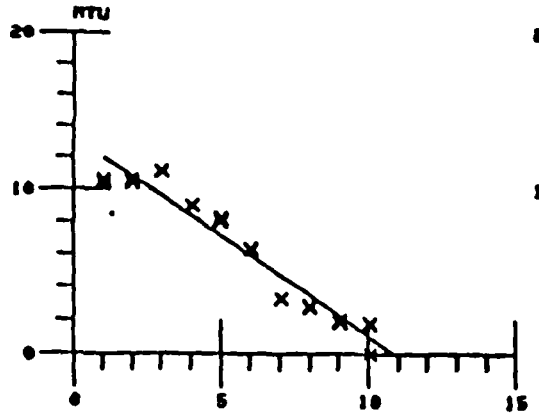
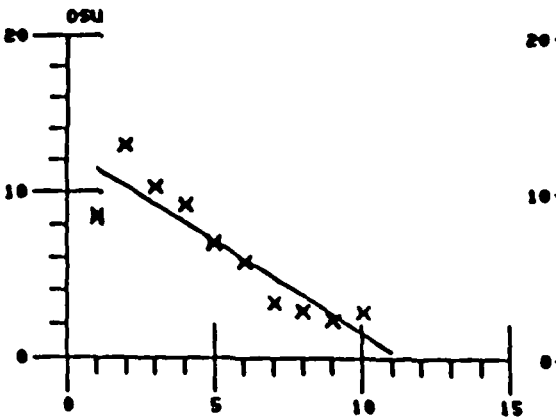

MTU
5.0
2.5
0.0
0
5
10
15
Y = 0.068 X + 2.888
YBAR = 3.184 SY = 0.266
SYX = 0.179 R = 0.739
OSU
Y = 0.045 X + 3.413
YBAR = 3.660 SY = 0.348
SYX = 0.319 R = 0.357
HAEC(OLYMPUS)
Y = -0.050 X + 3.884
YBAR = 3.608 SY = 0.400
SYX = 0.376 R = 0.338
FOXBORO
Y = 0.061 X + 3.320
YBAR = 3.654 SY = 0.300
SYX = 0.246 R = 0.575
JOAP-TSC
Y = 0.067 X + 3.185
YBAR = 3.484 SY = 0.301
SYX = 0.238 R = 0.636
PHASE II STAGE II MTI PN887
FERROGRAPHY DATA
DENSITY VS SLIDE POSITION
MTU MINERAL

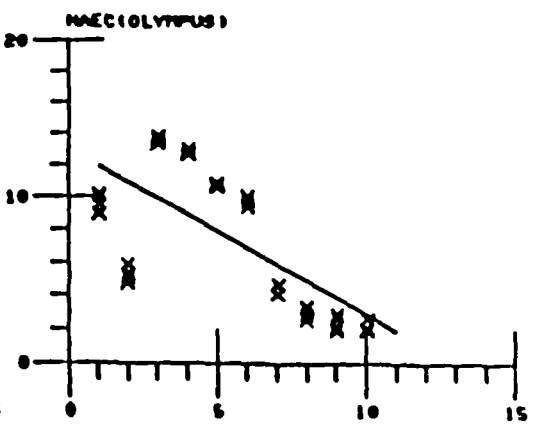
FIGURE 56



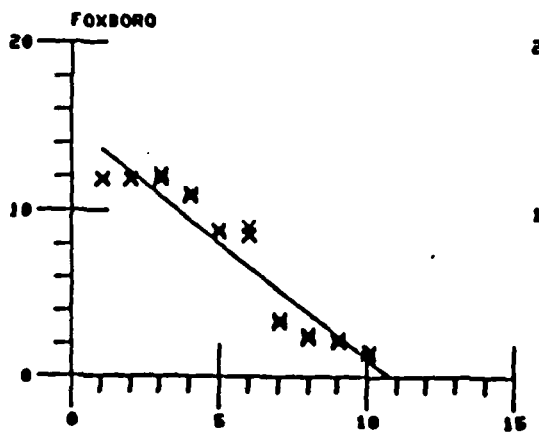
Y = -1.224 X +13.221
YBAR = 6.498 SY = 3.690
SYX = 1.012 R = 0.962



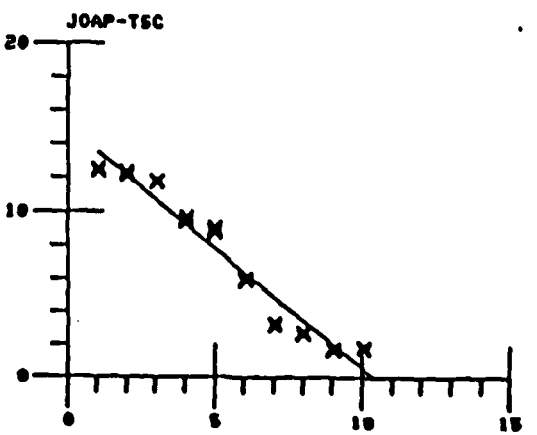
Y = -1.114 X +12.596
YBAR = 6.470 SY = 3.582
SYX = 1.562 R = 0.900



Y = -0.999 X +12.921
YBAR = 7.428 SY = 4.216
SYX = 3.894 R = 0.679



Y = -1.396 X +15.056
YBAR = 7.278 SY = 4.857
SYX = 1.284 R = 0.960



Y = -1.438 X +14.965
YBAR = 7.644 SY = 4.877
SYX = 0.947 R = 0.976

PHASE II STAGE II NTI PH887

FERROGRAPHY DATA

DENSITY VS SLIDE POSITION

OSU HYDRAULIC

FIGURE 57

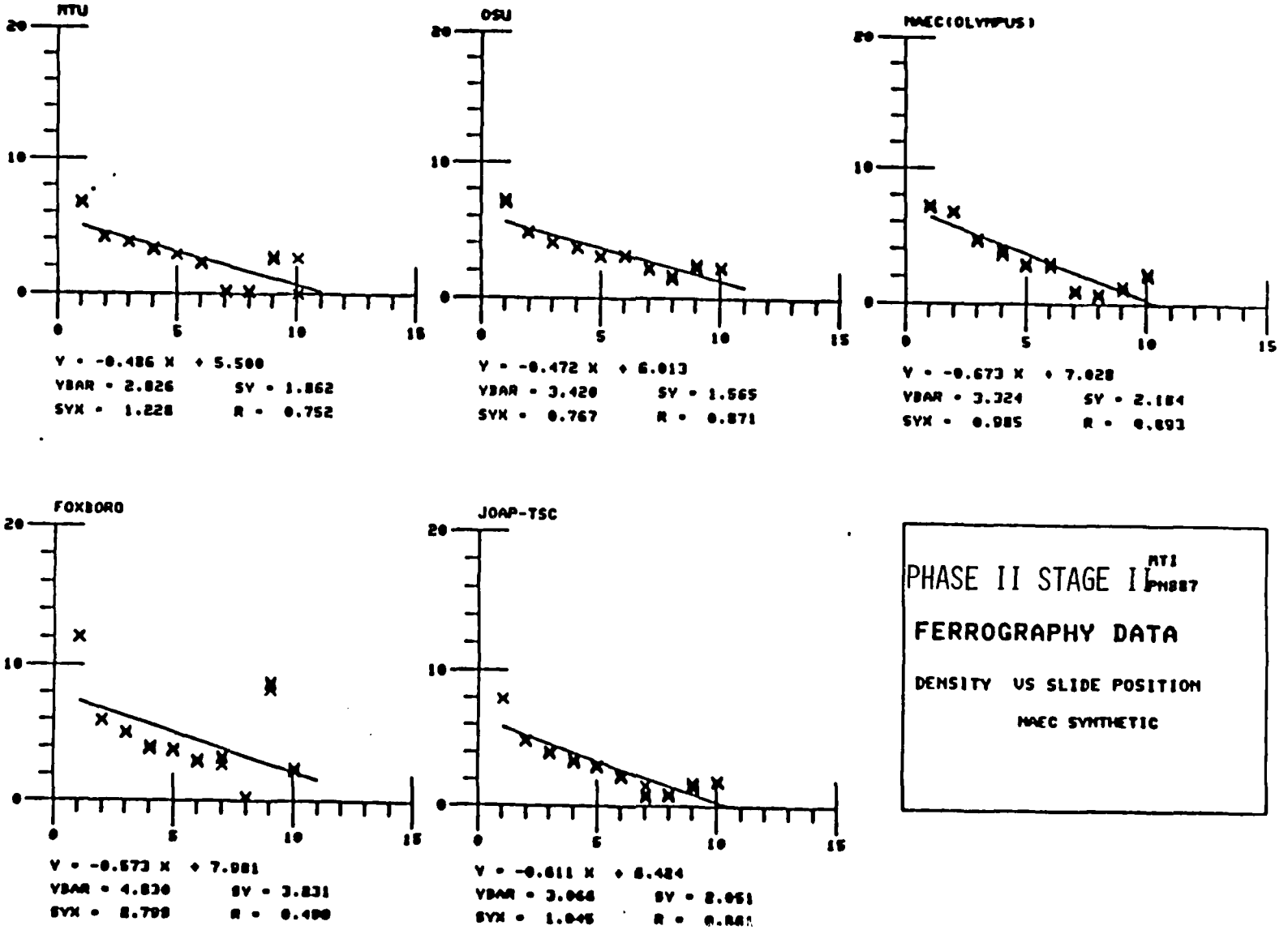
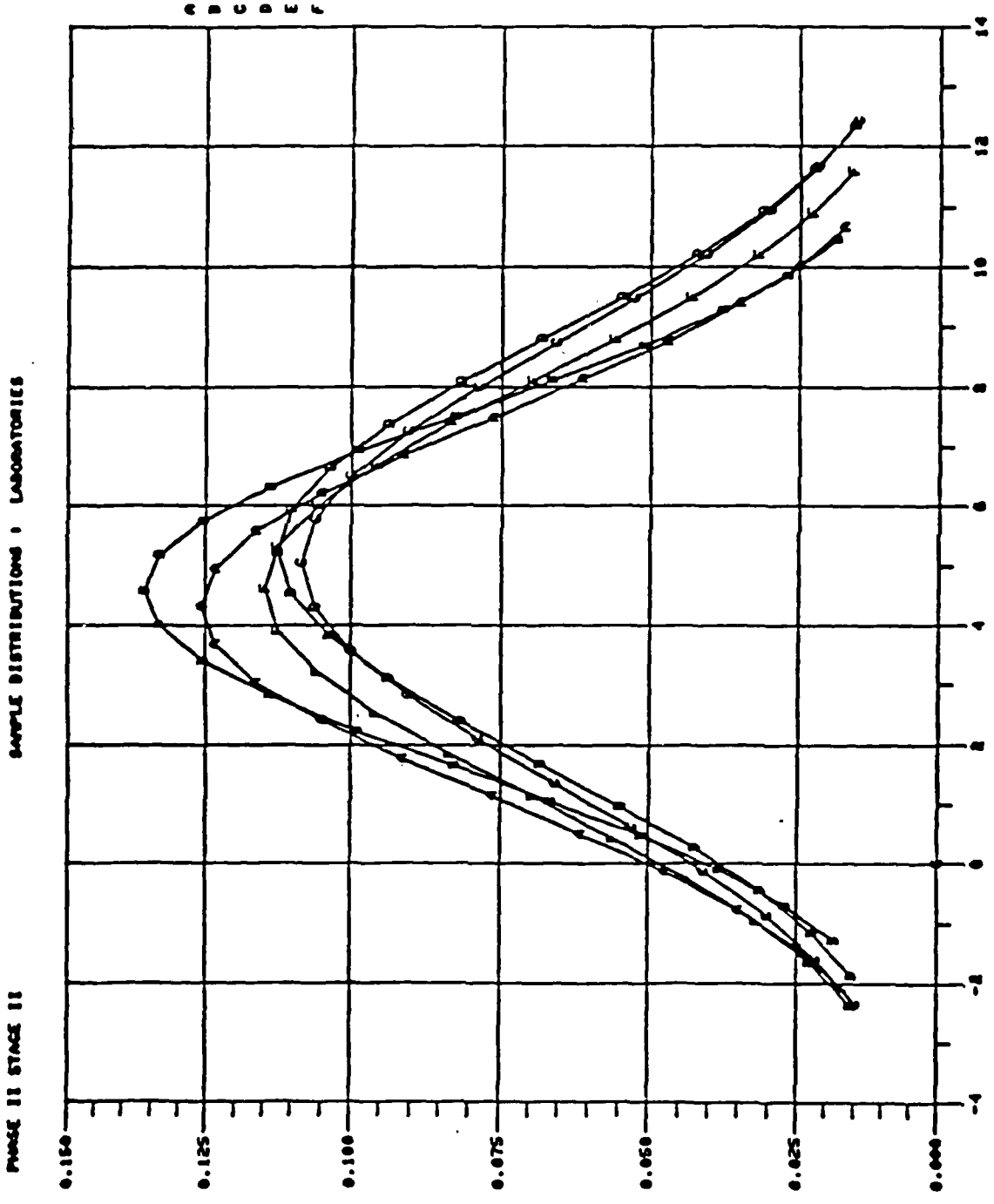

RTU
Y = -0.486 X + 5.500
YBAR = 2.826 SY = 1.862
SYX = 1.228 R = 0.752
OSU
Y = -0.472 X + 6.013
YBAR = 3.420 SY = 1.565
SYX = 0.767 R = 0.871
MAEC(OLYMPUS)
Y = -0.673 X + 7.028
YBAR = 3.324 SY = 2.184
SYX = 0.985 R = 0.893
FOXBORO
Y = -0.573 X + 7.981
YBAR = 4.830 SY = 3.831
SYX = 8.799 R = 0.490
JOAP-TSC
Y = -0.611 X + 8.484
YBAR = 3.066 SY = 8.051
SYX = 1.045 R = 0.881
PHASE II STAGE II
RTI
PN887
FERROGRAPHY DATA
DENSITY VS SLIDE POSITION
MAEC SYNTHETIC

FIGURE 58


PHASE II STAGE II
SAMPLE DISTRIBUTIONS - LABORATORIES
A - RTU
B - OSU
C - MARCIOLYMPUS)
D - FOXBORO
E - MARCIREICHERT)
F - JOAP-TSC
0.150
0.125
0.100
0.075
0.050
0.025
0.000
-4
-2
0
2
4
6
8
10
12
14

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ANALYTICAL FERROGRAPHY STANDARDIZATION.(U)
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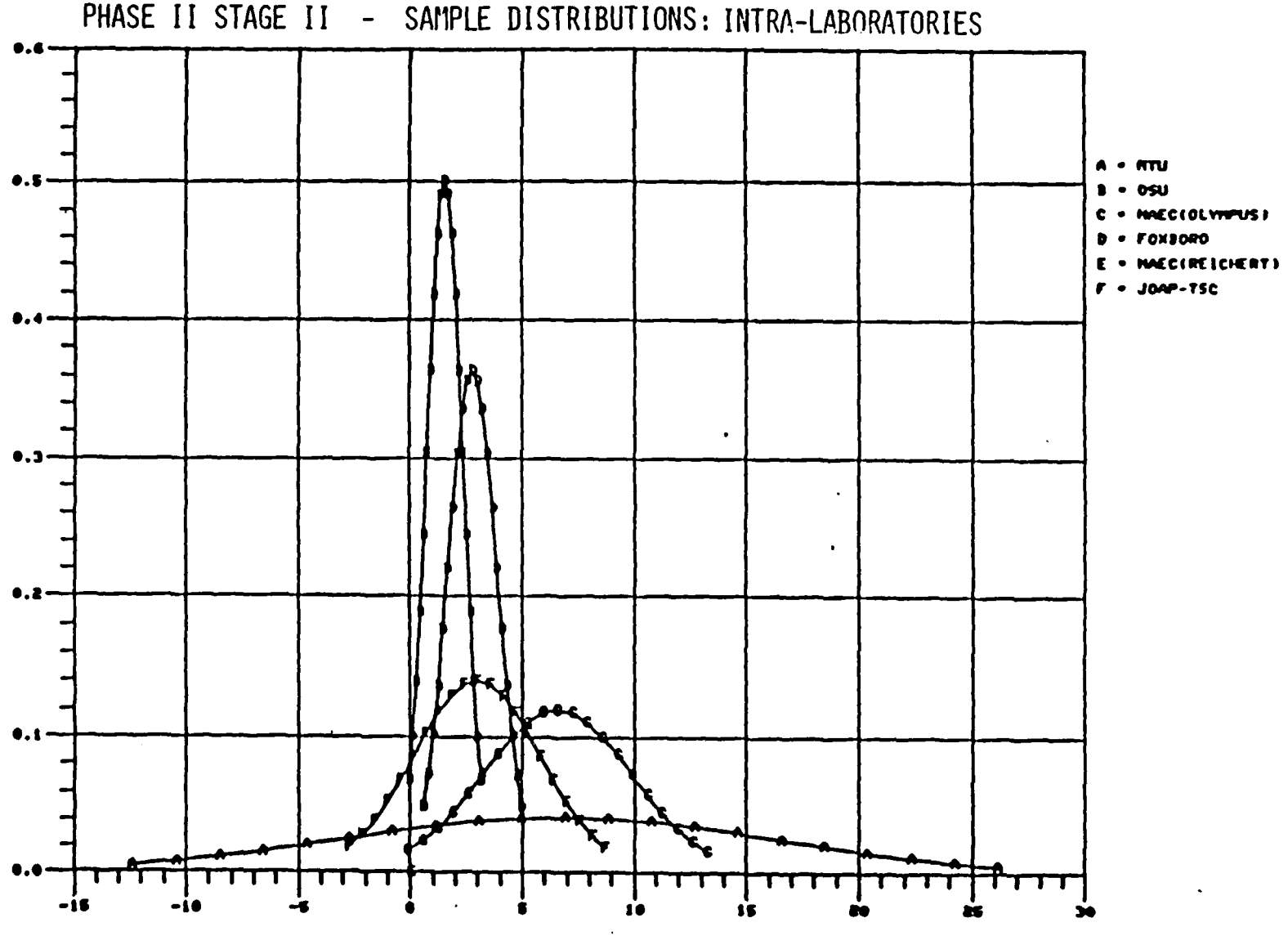
FIGURE 59


PHASE II STAGE II - SAMPLE DISTRIBUTIONS: INTRA-LABORATORIES
0.6
0.5
0.4
0.3
0.2
0.1
0.0
-15
-10
-5
0
5
10
15
20
25
30
A = RTU
B = OSU
C = MAEC(OLYMPUS)
D = FOXBORO
E = MAEC(REICHERT)
F = JOAP-TSC

2) Sample Type as presented in Figure 60

(a) Slight variation between types of fluid.

3) Slide Position

(a) COV consistent with respect to slide position.

B. Interlaboratory as presented in Table 5

1) Range $\simeq$ 10 - 98%

Mean $\simeq$ 41%

4.4.3 Graphical Regression Analysis

Representative Phase II - Stage II regression analysis plots are presented in Figures 61, 62, and 63.

The following results can be drawn from the analysis of the total regression analysis plot population.

A. Poor quantitative interlaboratory correlation exists

B. Inconsistent data bias exists.

C. Poor discrimination as presented in Figure 64

Mean $\simeq$ 2.3

D. Substantial scatter exists.

FIGURE 60

PHASE II STAGE II

COEFFICIENT OF VARIATION
SAMPLE DISTRIBUTIONS: TYPES OF OIL

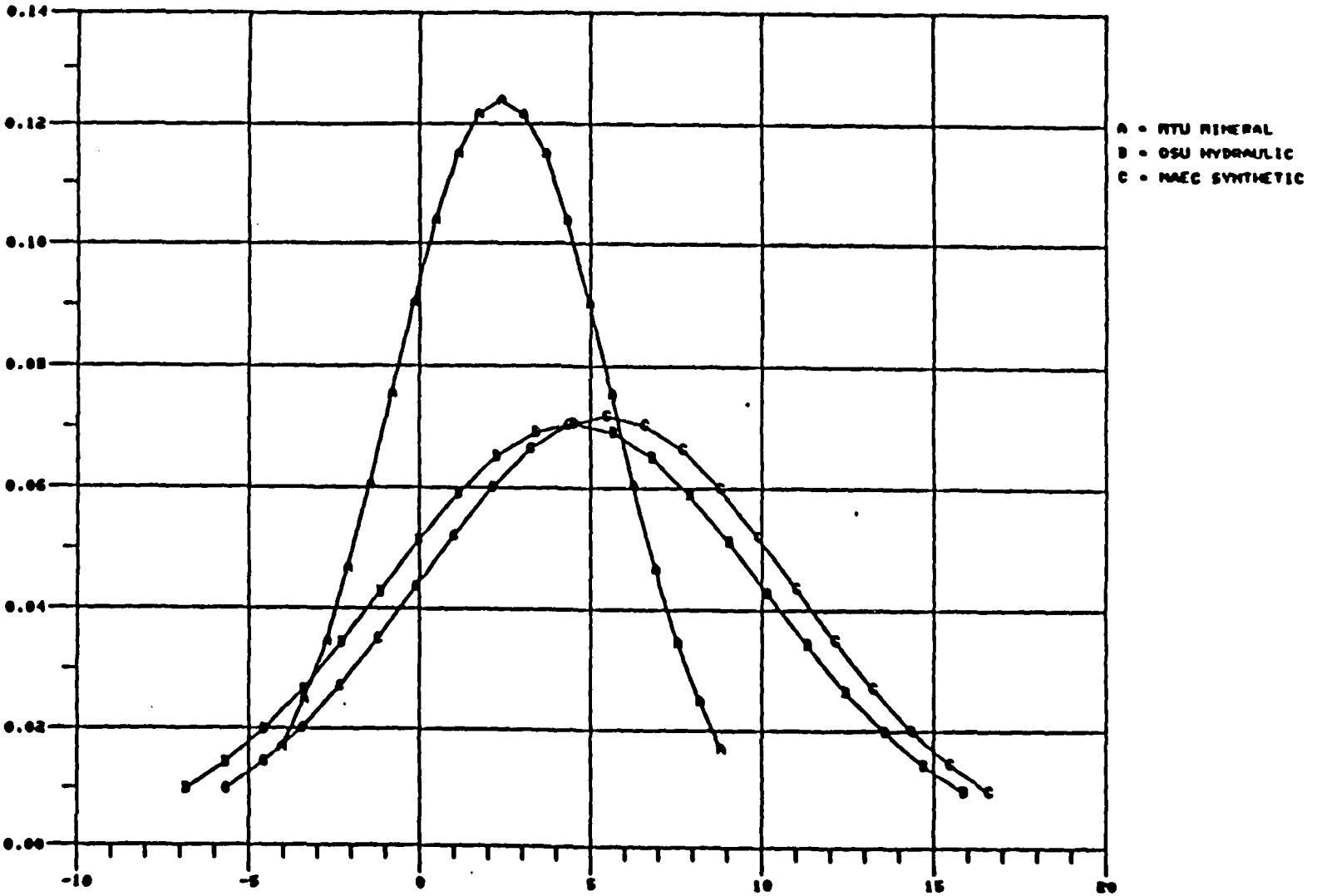


TABLE 5

PHASE II STAGE II - COEFFICIENT OF VARIATIONS INTERLABORATORY

OIL	CON	LAB	COEFFICIENTS OF VARIATION			AVG.FOR SLIDE
1	0	1	0.0	0.0	0.1309E+01	0.4362E+00
1	0	1	0.0	0.1732E+01	0.1584E+00	0.6300E+00
2	0	1	0.6735E-01	0.4927E+00	0.1509E+01	0.6898E+00
3	0	1	0.7532E+00	0.1745E+01	0.0	0.8327E+00
2	0	1	0.1257E+01	0.1411E+01	0.5602E+02	0.1956E+02
3	0	1	0.1060E+01	0.0	0.5590E+02	0.1899E+02
2	0	4	0.8753E+00	0.4172E+00	0.1223E+02	0.4509E+01
2	0	4	0.4704E+00	0.1025E+01	0.5653E+01	0.2383E+01
1	0	4	0.5503E+01	0.3458E+01	0.1256E+01	0.3406E+01
1	0	4	0.1595E+01	0.9397E-01	0.2479E+01	0.1389E+01
3	0	4	0.4567E+00	0.2115E+01	0.5440E+01	0.2671E+01
3	0	4	0.3845E+00	0.1862E+01	0.3791E+01	0.2013E+01
2	0	6	0.5625E+00	0.1546E+01	0.3670E+01	0.1926E+01
2	0	6	0.3650E+00	0.1782E+01	0.4700E+01	0.2282E+01
1	0	6	0.0	0.2246E+01	0.1430E+01	0.1225E+01
1	0	6	0.0	0.2019E+01	0.1292E+01	0.1104E+01
3	0	6	0.1851E+01	0.8619E+01	0.1547E+02	0.8648E+01
3	0	6	0.9889E-01	0.2808E+01	0.4450E+01	0.2452E+01
2	0	2	0.1913E+01	0.1241E+00	0.2053E+01	0.1363E+01
2	0	2	0.1063E+01	0.1211E+01	0.0	0.7582E+00
1	0	2	0.1407E+00	0.0	0.1428E+01	0.5228E+00
1	0	2	0.1643E+01	0.1464E+01	0.1731E+01	0.1613E+01
3	0	2	0.1884E+01	0.2979E+01	0.2342E+01	0.2401E+01
3	0	2	0.2087E+01	0.1483E+01	0.3670E+01	0.2413E+01
1	0	3	0.1145E+02	0.1264E+02	0.9374E+01	0.1116E+02
1	0	3	0.2829E+01	0.2448E+01	0.2011E+01	0.2429E+01
3	0	3	0.3653E+01	0.6299E+01	0.1838E+02	0.9446E+01
3	0	3	0.1821E+01	0.4877E+01	0.7187E+01	0.4628E+01
2	0	3	0.2026E+01	0.1753E+01	0.9117E+01	0.4299E+01
2	0	3	0.6098E+01	0.1415E+01	0.1424E+02	0.7252E+01

FOR 30 SAMPLES, THE AVERAGE COEFFICIENT OF VARIATION IS 0.4114E+01
WHILE THE STANDARD DEVIATION IS 0.4945E+01

FIGURE 61

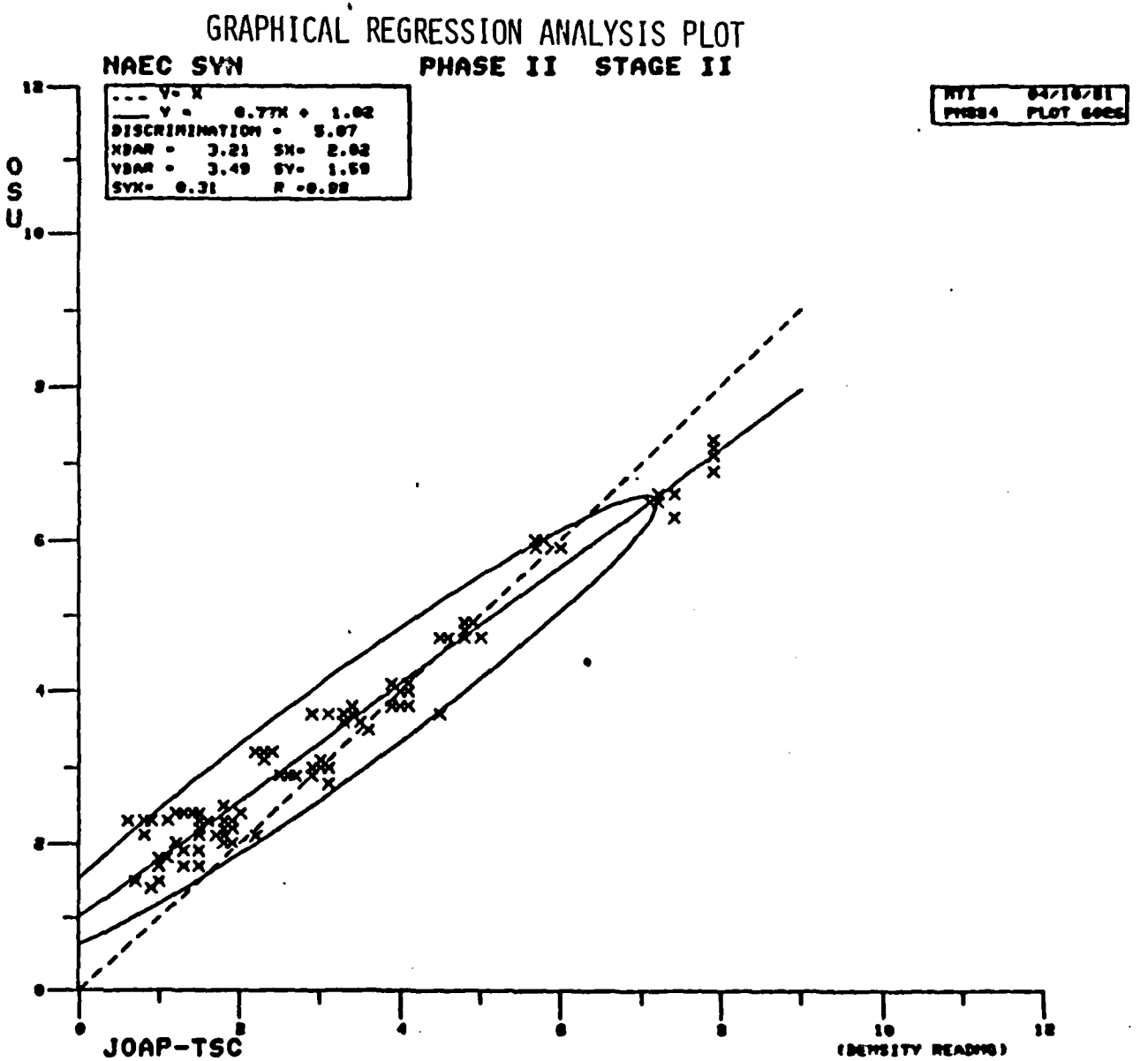

GRAPHICAL REGRESSION ANALYSIS PLOT
NAEC SYN
PHASE II STAGE II
--- V= X
—— V = 0.77X + 1.02
DISCRIMINATION = 5.07
XBAR = 3.21 SX= 2.02
YBAR = 3.49 SY= 1.59
SYX= 0.31 R =0.98
RTI 04/16/81
PHASE4 PLOT 6005
OSU
JOAP-TSC
(DENSITY READING)
0
2
4
6
8
10
12

FIGURE 62

GRAPHICAL REGRESSION ANALYSIS PLOT

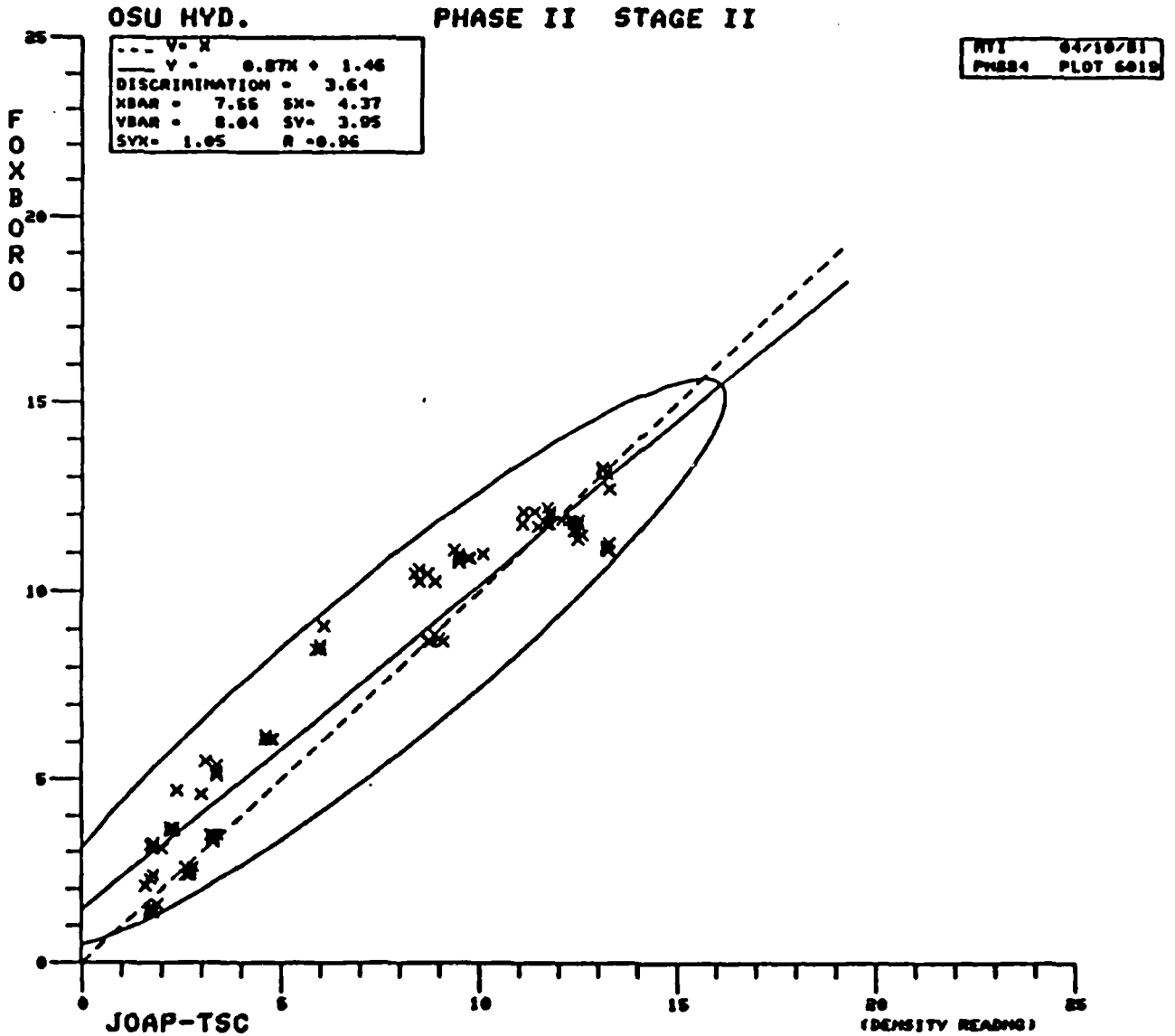


FIGURE 63

GRAPHICAL REGRESSION ANALYSIS PLOT

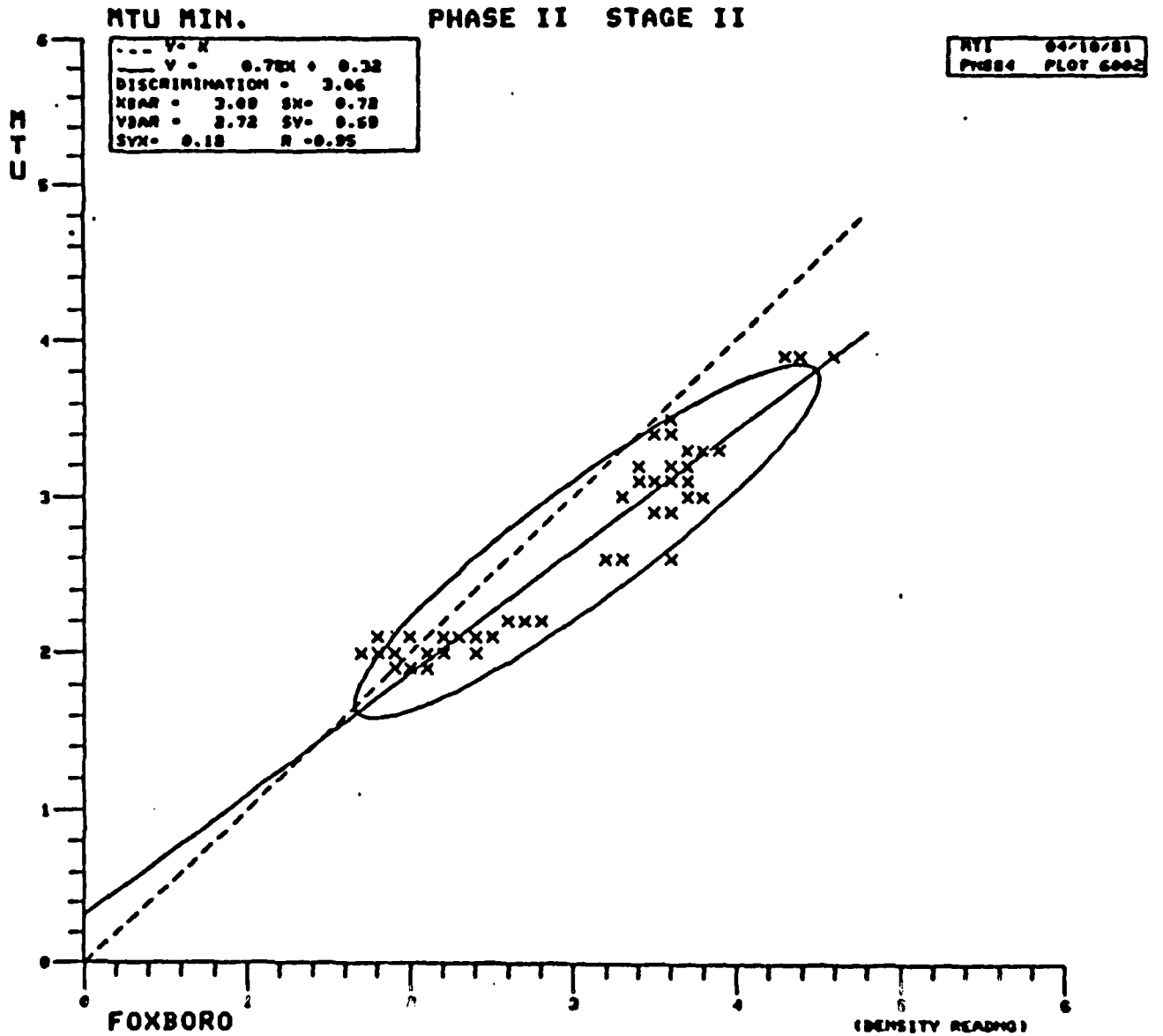
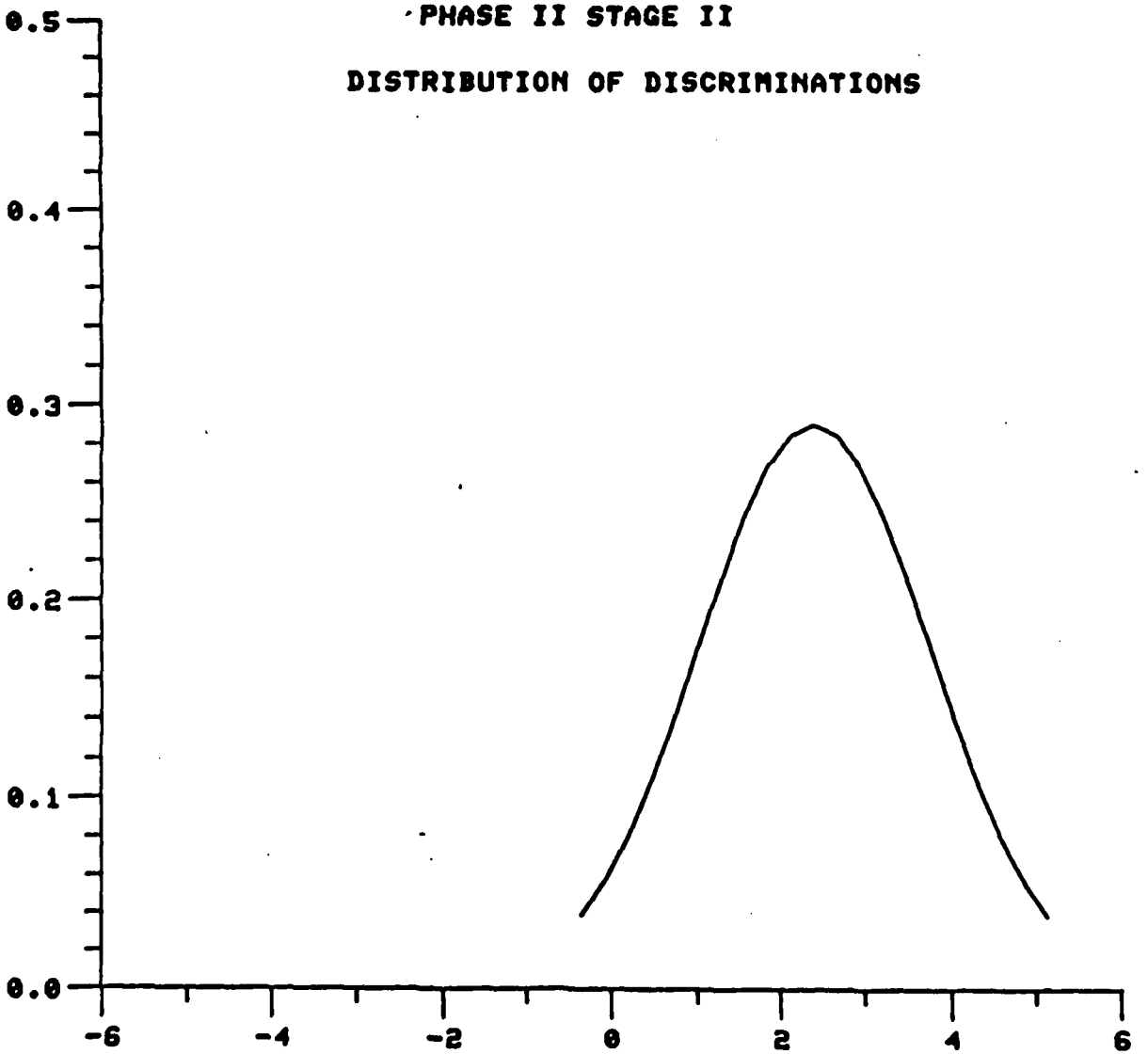


FIGURE 64


PHASE II STAGE II
DISTRIBUTION OF DISCRIMINATIONS
0.5
0.4
0.3
0.2
0.1
0.0
-6
-4
-2
0
2
4
6

5.0 PROGRAM STATISTICAL SUMMARY AND CONCLUSIONS

Based on the statistical results presented above for each program phase, the following summary and conclusions have been generated. These results and conclusions are presented with respect to the primary and secondary analysis variables as covered in Section 3.1.

5.1 Lighting Technique

Both transmitted and reflected lighting approaches were considered with respect to Analytical Ferrography density measurements.

A. Mean reflected density reading is higher than the transmitted mean reading.
B. Trends the same.
C. Standard deviation the same.
D. Coefficient of variation the same.
E. Standardize on reflected approach.

5.2 Indexing Technique

Both a fixed density reading slide indexing approach (conventional) and a floating slide indexing approach (new method) were considered.

A. Mean conventional density reading is the same as the new method mean reading.
B. Trends the same.
C. Standard deviation the same.
D. Coefficient of variation the same.
E. Standardize on conventional approach.

5.3 Equipment

Both Reichert and Olympus Ferroscopes were considered under this round robin effort.

A. Mean Reichert equipment density reading is the same as the Olympus mean reading.
B. Trends the same.
C. Standard deviation the same.
D. Coefficient of variation the same.
E. Minimal Ferroscope type effect on variation.

5.4 Slide Position

Repeatability with respect to the indexed location on the Ferrogram was considered. Debris size is a function of this slide location.

A. Minimal effect on variation which is somewhat of an unexpected result.

5.5 Sample Type

Hydraulic fluid, mineral oil, and synthetic lubricant samples were considered.

A. Minimal effect on repeatability.
B. Hydraulic samples consistently higher variation.

5.6 Sample Debris Concentration

Light, medium, and heavy debris sample concentrations were considered.

A. Light and heavy concentrations have relatively the greatest effect on variation.

5.7 Intra-Laboratory

Repeatability within each laboratory was considered.

A. Trends agree.
B. Good repeatability within each laboratory.

	Phase I Stage I	Phase II Stage I	Phase II Stage II
- Mean COV	19%	5%	4%

C. Procedure improved intra-laboratory variation.

5.8 Interlaboratory

Repeatability between all participating laboratories was considered.

A. Trends agree.

B. Poor repeatability between laboratories.

	Phase I Stage I	Phase I Stage II	Phase II Stage I	Phase II Stage II
- Mean COV	50%	57%	37%	41%

C. Poor Discrimination between laboratories.

- Mean Discrimination	.91	1.5	.84	2.3

D. Inconsistent interlaboratory bias.

E. Procedure has only limited effect on interlaboratory variation.

F. The prime source of interlaboratory variation appears to be the Ferroscope densitometer as opposed to either the operator or the sample procedure variables.

5.9 Summary

The results of this analysis have served to indicate that the draft Navy Analytical Ferrography Standardized Procedure has served to greatly improve the repeatability of Analytical Ferrographic analysis within an individual laboratory. However, repeatability between laboratories remains poor due to what appears to be an equipment problem with the Ferroscope densitometer. This problem could be addressed through the development of an effective densitometer calibration standard.

These results should not be construed as an effectiveness criticism of either Ferrographic Analysis or wear debris analysis technology. Both of these interrelated technical fields have proven their effectiveness in both the maintenance and research communities. Care should be exercised however, when comparing/correlating Ferrography results from different laboratories/equipments.

6.0 NAVAL ANALYTICAL FERROGRAPHY STANDARDIZED PROCEDURE

Based on wear particle analysis research, Ferrography experience, statistical analysis, and laboratory inputs, a draft Analytical Ferrography Standardized Procedure has been developed under this program. The resultant draft procedure is presented in Appendix A of this report.

As discussed earlier in this report, the standardized procedure will result in analysis repeatability exhibiting a mean coefficient of variation within a laboratory of approximately 5%. However, this procedure does not guarantee adequate interlaboratory analysis repeatability due to overriding equipment variations as presented in Section 5.9.

7.0 NAVAL FERROGRAPHY VERIFICATION PROGRAM RECOMMENDATIONS

Verification program analysis and conclusions have identified several areas warranting further attention. In light of these identified areas, the following short term and long term recommendations are presented.

7.1 Short Term Recommendations

A. Publish Naval Analytical Ferrography Standardized Procedure.

B. Establish the procedure as a technical standard through an appropriate standards organization.

C. Pursue Analytical Ferrography Densitometer calibration technique.

D. Verify Analytical Ferrography Densitometer calibration technique.

E. Develop a Direct Reading Ferrography standardized procedure.

F. Verify direct Reading Ferrography repeatability.

7.2 Long Term Recommendations

A. Develop a comprehensive, repeatable, precise wear particle characterization approach/technique/equipment. This new characterization approach should assess lubricant borne debris concentration, size distribution, composition, and morphology for both metallic and nonmetallic particles.

8.0 REFERENCES

1. Wescott, V. C., et al, "Oil Analysis Program," U.S. Naval Air Engineering Center, Final Report on Contract Number N00156-74-C-1682, Foxboro-Trans Sonics Report, 1975.

2. Scott, D., and Seifert, W. W., "Ferrography - A New Tool For Analyzing Wear Conditions," Fluid Power Testing Symposium, 1976.

3. Dalah, H., and Senholzi, P. B., "Characteristics of Wear Particles Generated During Failure Progression of Roller Bearings," ASLE Paper presented at ASLE Annual Meeting, 1976.

4. Senholzi, P. B., and Bowen, C. R., "Oil Analysis Research," National Conference on Fluid Power, October 1976.

5. Senholzi, P. B., "Oil Analysis/Wear Particle Analysis," Mechanical Failures Prevention Group, 1977.

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8. Naval Air Engineering Center Report No. 92-0458, "Sample Preparation/ Ferrogram Procedure/Ferrogram Analysis," August 8, 1980.

9. Naval Air Engineering Center Tribology Laboratory Ferrogram Analysis Report, November 7, 1977.

10. Bowen, E. R., and Westcott, V. C., "Wear Particle Atlas," Contract No. N00156-74-C-1682, U.S. Naval Air Engineering Center, Controlling Office, July 1976.

APPENDIX A

INTERIM DRAFT

NAVAL
ANALYTICAL FERROGRAPHY
STANDARDIZED PROCEDURE

MARCH 1982

Sponsor:

Office of Naval Research

Mechanical Technology Incorporated
1656 Homewood Landing Road
Annapolis, Maryland 21401

1. SCOPE

1.1 This method covers the evaluation of liquid borne ferrous and ferromagnetic wear debris particulate by means of a magnetic separation technique. It is applicable to mineral and synthetic lubricants as well as other viscous fluids.

1.2 This method provides for the preparation and density determination of the wear debris deposited on a glass substrate. A calculation procedure for normalization of density data is provided.

NOTE 1: This method also has been applied to the analysis of wear debris contained in grease. However, sufficient data is not available to include this procedure here.

2. SUMMARY OF METHOD

2.1 A fluid sample is pumped over a prepared glass substrate located in a high gradient magnetic field. The ferrous and ferromagnetic wear debris is deposited on the glass substrate according to size and a determination of the relative density of the particulate along the slide length is measure.

NOTE 2: Mechanical trapping and gravitational effects will influence liquid borne non-ferrous metallic and hybrid wear debris. Although the deposition of these particles will not be accurate with respect to size on the substrate, their presence should be noted.

3. DEFINITIONS

3.1 Density - Percent light reduction which is a function of the particle concentration at various locations on the substrate.

3.2 Significant deposit - The concentration (density) of particles nearest the entry point of the substrate, normally exhibiting the largest percent of light reduction (approximately 55 mm up from the exit end of the slide).

3.3 Percent Area Covered - Used in quantitative analysis, the percent of the area covered by large ferrous particles (A_L) and small ferrous particles (A_S), usually located at the entry area (55 mm) and 50 mm locations, respectively.

4. SIGNIFICANCE AND USE

Ferrographic analysis provides an assessment of wear debris quantity, size distribution, composition, and morphology. This analysis is useful in assessing the wear condition of a lubricated component/system.

5. APPARATUS

5.1 Analytical Equipment

5.1.1 Fluid Analyzer, either Model 7058-3 (dual) or Model 7069-4 (duplex).

5.1.2 Ferroscope, Model 7507-7 or equivalent microscope, having both transmitted and reflected light capability.

5.1.3 Density Reader, Model 7079-3 with optical measurement device and digital readout.

5.1.4 Oven or equivalent heating device, capable of maintaining constant temperature at 150°F (65.5°C) ± 3.

NOTE 3: With the exception of 5.4, the only recognized and licensed distributor of the equipment described is Foxboro Analytical, Burlington, Massachusetts 01803.

5.2 Analytical Ferrograph Materials

5.2.1 Fixer/Solvent - Filtered tetrachloroethylene (Regent Grade)

5.2.2 Delivery Tubing - Teflon, 1/16 in. (1.58 mm) I.D., cut to lengths of 25 in. (635 mm).

5.2.3 Substrate - Microcover glass, 24 mm x 60 mm, thickness approximately .20 mm, with non-wetting barrier (Nyebar) applied.

5.2.4 Vials - Pre-cleaned, clear glass, 15 ml capacity, with caps.

5.2.5 Pipette Dispenser - 1 ml pre-calibrated, disposable pipettes.

NOTE 4: The Analytical Ferrograph materials can be obtained directly from Foxboro Analytical, Burlington, Massachusetts 01803.

NOTE 5: The fixer (tetrachlorethylene) is a nonflammable chlorinated hydrocarbon. Avoid skin contact and use only in well ventilated area.

NOTE 6: It is necessary that the evaluator fully understand the operation and general operating procedures of this equipment.

6. PREPARATION OF APPARATUS - ANALYZER

6.1 Remove delivery tubing from protective bag and cut one end at a 45° angle to the axis of the tubing. Use a sharp instrument (razor or scalpel blade) as opposed to scissors.

NOTE 7: If only a qualitative analysis is desired, the tube should not be cut on a 45° angle. This allows the particles to spread out, facilitating morphological identification.

6.2 Position the delivery tube in the analyzer turret arm with the 45° end extending approximately 1/8 in. (3.17 mm) beyond the tip of arm. (See Figure 1).

6.3 Thread the tubing through pump exit tubing clamp, pump delivery arms, and pump entrance clamp and lightly secure delivery arms.

6.4 Remove glass substrate from protective bag and insert into substrate fixture appropriately retracting and releasing positioning pin. Determine that the substrate is properly positioned on the metal shelf, located at the top of the slide bed. The "closed loop" end of the barrier on the substrate should be located closest to the turret arm. A black dot on the slide should be located at lower left hand corner of slide (plain view) (see Figure 2).

6.5 Position the turret arm so that the exit end of the supported tubing is slightly above the surface of the slide. (Avoid dripping of sample on the slide).

6.6 Rotate the drain tube holding fixture counterclockwise and lower the notched end of tube until it resets on the exit edge of the slide.

7.0 SAMPLE PREPARATION

NOTE 8: Samples should be stored at 0°F (-17.8°C) if ferrographic analysis is not performed within 24 hours of sampling. If stored under these conditions, sample should be heated to 185°F (85°C) for thirty (30) minutes prior to step 7.1.2.

7.1 Undiluted Sample

7.1.1 Shake sample bottle by hand vigorously for approximately one (1) minute. Loosen sample bottle cap (do not remove) and place in suitable heating apparatus. Raise temperature of the fluid to 150°F (65.5°C) and maintain such temperature for a period of ten (10) minutes.

7.1.2 Pipette 1 ml of fixer solution into a clean mixing vial.

7.1.3 Remove sample bottle from heating apparatus and tighten cap. Hand shake sample fluid vigorously for sixty (60) seconds. Pipette 3 ml of sample fluid into same mixing vial, cap, and hand shake for ten (10) seconds.

8.6 Upon completion of the sample mixture pumping cycle reset pump timer to 14 minutes and initiate a fixer pumping cycle. During this cycle, approximately three (3) air bubbles should be intermittently introduced into the delivery tube. These bubbles are created by removing and reinserting the delivery tube into the fixer solution. Space air bubbles approximately five (5) seconds apart.

8.7 Upon completion of the fixer pumping cycle lift the turret arm/delivery tubing from the glass substrate and allow the slide to drain off any remaining fluid.

8.8 Allow the slide to drain off any remaining fluid.

8.8 Allow the slide to dry completely before removing from the analyzer (approximately (20) twenty minutes). Institute measures to avoid air-borne contaminants.

8.9 Remove glass substrate vertically from analyzer and affix identification tag. Place in protective cover until ready for analysis.

8.10 Discard all materials (pipette tips, vials, and tubing) used in making slide. Reuse may introduce contaminants from previous samples, thereby introducing error into the analysis.

9.0 PREPARATION OF APPARATUS - DENSITY READER

9.1 Prepare microscope and density reader according to the procedure below. (Allow at least 30-45 minutes for initial warm-up of density reader.)

NOTE 12: Room temperature, in which equipment is operated, should be 75°F (21°C), to avoid overheating density reader, resulting in erratic behavior.

9.1.2 In order to adjust the reader for full scale, keep the light path selector lever fully in and adjust thepotentiometer in the rear of the reader to give a 100% area covered reading.

9.1.3 To zero the reader, the following procedures must be followed.

9.1.3.1 Using ordinary visual reflected light and the 10x objective, locate an area outside the barrier on the ferrogram slide that is free of any particles. Withdraw the light path selector lever and hold the reflected light switch on read.

9.1.3.2 Attempt to zero the reader by adjusting the potentiometer on the front of the reader. If this cannot be achieved, adjust the regulated voltage supply to the reflected lamp by using the potentiometer at the rear of the power supply. The light intensity may be further reduced by closing down the aperture diaphragm.

9.1.3.3 By the adjustment of both potentiometers and the aperture diaphragm the reader should be able to be zeroed.

10.0 OPERATING PROCEDURE - DENSITY READINGS

10.0 Density readings are taken on the Ferrogram in order to assess the relative quantity of debris at varous locations along the length of the substrate. The percent area covered by debris (density) is determined from an approximate 1 mm diameter field of view. This relative measure of debris can be normalized to a unit volume of actual used sample oil over the Ferrogram. (trending of data over a series of oil samples) and for interlab comparisons. Note that the percent area covered refers to the light reflected by the debris on the Ferrogram in the field of view.

10.2 Place slide on microscope stage with the entry deposit of the slide to the evaluator's left. Using a 10x objective, locate the significant deposit of particles in the following manner.

10.2.1 Locate the densest part of the significant deposit and center in the field of view. (See Figure 3). With the reflected light switch held on read, operate the x and y controls of the stage to obtain the maximum values.

NOTE 13: It is recommended that the average of three readings per each longitudinal location be recorded.

NOTE 14: Verification efforts have revealed that the lighting approach (transmitted or reflected) has no statistical significance. However, some users have found the elimination of stray light (room lighting) has stabilized their readings when operating in the reflected mode.

10.2.2 Record the longitudinal location (57 mm, 56 mm, etc.) from the indexing scale located on the microscope stage.

10.2.3 In order to insure a credible density reading a range of 10-40% must be maintained. If the significant deposit exceeds 40% density then the sample requires dilution. (See step 7.2). If the deposit falls below a 10% reading, then the volume of sample oil must be adjusted accordingly.

10.3 Using the same 10x objective, take a photo of the signifcant deposit for comparison purposes.

10.4 For the remainder of the slide, traverse the debris track slowly, recording the maximum reading for any select position. Carefully record the values with respect to the index location.

11.0 DATA HANDLING AND CALCULATIONS

11.1 Record pertinent data for all aspects of the analysis. This data includes:

A. Ferrogram Numbers
B. Oil Sample Identification
C. Date of Analysis
D. Name of Evaluator
E. Volume of Fixer used in Mixture
F. Volume of Sample Oil in Mixture
G. Volume of Dilution Oil (if any)
H. Density Reading (Raw Data) Per Position
I. Normalized Density Reading Per Position

11.2 Following completion of all density reading, the raw data generated must be normalized (percent per ml) utilizing the following formula:

$$A_N = \frac{A}{V_T \times \frac{V_O V_O}{V_O + V_F}}$$

where:

A_N = density reading normalized to 1 ml of sample (dirty) oil
A = raw density reading from ferrogram
V_T = total volume fluid pumped over ferrogram (from mixer vial)
V_O = volume oil in mixer vial
V_F = volume fixer in mixer vial

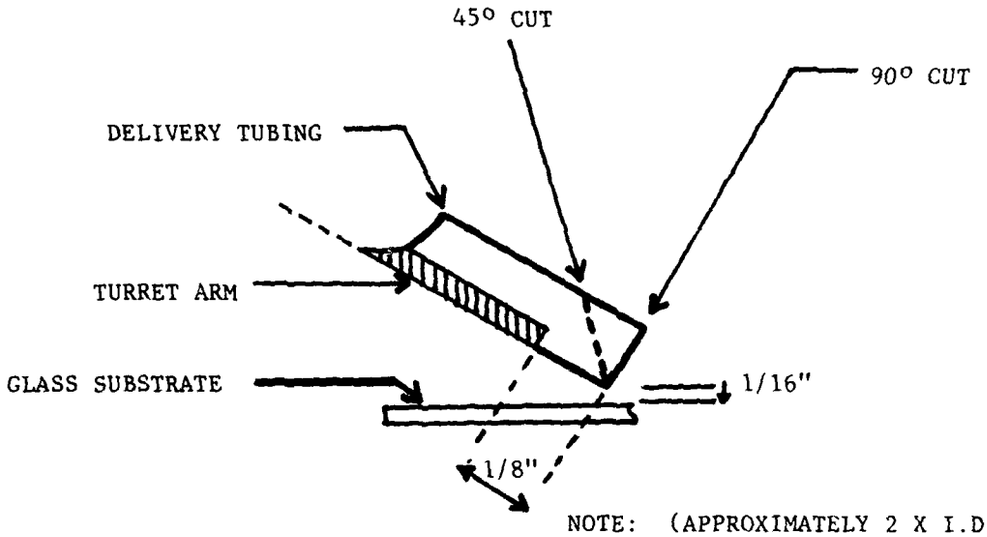
11.3 If dilution procedures were performed, the following formula should be used:

$$A_N = \frac{A}{V_T \times \frac{V_O}{V_O + V_F} \times \frac{V_S}{V_S + V_D}}$$

where:

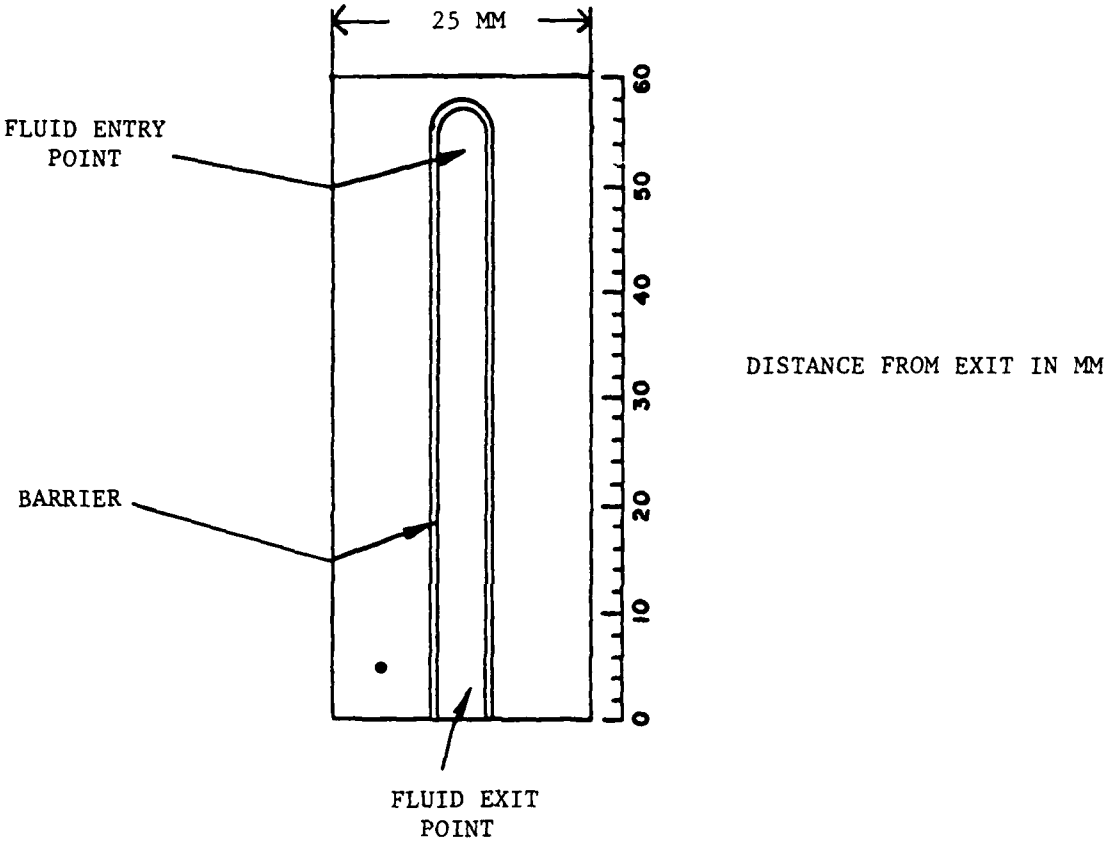
V_S = volume of sample oil used in dilution procedure
V_D = volume of clean diluent oil used in the dilution procedure

NOTE 15: Normalized density data should be reported to one decimal place (i.e. 3.9, 12.6, etc.).



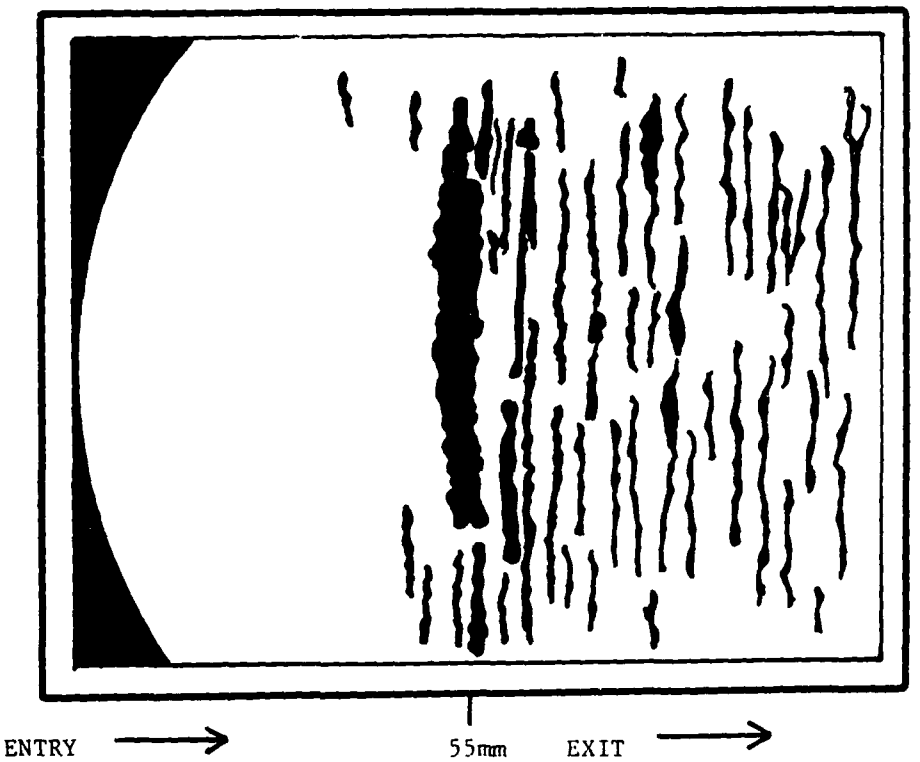
DELIVERY TUBING PREPARATION AND POSITIONING

FIGURE 1



FERROGRAM SLIDE DIMENSION AND KEY FACTORS

FIGURE 2



SIGNIFICANT DEPOSIT CENTERING
(MAGNIFICATION 100X)

FIGURE 3

ACKNOWLEDGEMENTS

The following people have contributed to this document:

Mr. M. P. O'Donnell, Naval Air Engineering Center
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Mr. C. Hubert, Michigan Technological University
Mr. C. P. Merhib, Army Materials and Mechanics Research Center
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Mr. D. Norvelle, Oklahoma State University
Ms. J. Dobson, Oklahoma State University
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Dr. W. Ruff, National Bureau of Standards
Mr. R. Lee, Joint Oil Analysis Program, Technical Support Center
LCDR H. P. Martin, Office of Naval Research

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